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Challenges in cleaning microstructured devices

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A B S T R A C T

Continuous crystallization of lipid nanoparticles, carrying bioactive compounds, is employed as a model system for food products to demonstrate benefits, application possibilities and challenges of microscale process engineering. It could be shown, that high and well-defined cooling rates (up to 10^4 times higher than standard batch processes) can be achieved by using a micro heat exchanger device for the continuous melt crystallization of lipid nanoparticles distributed in an emulsion, resulting in well-defined product qualities. Several formulations led to fouling and blocking of small passages in the micro heat exchanger. Fouling, blocking and associated challenging cleaning issues are often drawbacks for the use of micro devices for particulate flows in industry applications. Furthermore, no standardized design methods for cleaning strategies exist for microstructured devices.

This contribution addresses the design of cleaning strategies for microstructured devices, demonstrated using a model process. The design of the cleaning strategies at the micro scale is realized with standardized macroscopic methods, namely fluid dynamic gauging and a simple flow channel. Analogies and differences between cleaning macro and micro devices are discussed.

Keywords: Micro heat exchanger; Process intensification; Fouling; Cleaning of micro dimensions; Fluid dynamic gauging; lipid nanoparticles; crystallization

1. Introduction

Process intensification by the application of microscale process engineering, characterized by at least one performance relevant dimension being ≤1000 μm leads to the possibility, in reaction and heat transfer processes, of moving from batch to continuous processing mainly due to enhanced heat and mass transfer (Bayer et al., 2004; Hessel, 2009; Matsuyama et al., 2011). These effects are primarily caused by the very high surface-to-volume ratio in the microscale devices. Further advantages, particularly suitable for sensitive products, are the low shear stress levels (flow is typically in the laminar regime) and short residence times. In addition, the time-to-market can be drastically reduced by the use of micro devices for products with annual capacities lower than 100 tonnes (Grundemann et al., 2012). These characteristics fit very well with the requirements of the food industry to create functional foods for the delivery of bioactive food components, for example by the application of lipid nanoparticles (LNPs) (Weiss et al., 2008).

Schoenitz et al. (2014a) demonstrated that continuous crystallization of LNPs could be achieved in a micro heat exchanger. Furthermore, a subsequent microstructured device allowed for the crystal polymorphic form to be controlled.

Besides these benefits, application of micro structured devices for particulate flows is challenging, resulting in only occasional usage of these devices in industrial applications. Complete blocking of the microchannels can restrict the performance of these devices. Additionally, investigations of fouling phenomena and related cleaning of microstructured devices are rarely reported (Schoenitz et al., 2014b).

This contribution addresses the transfer of knowledge from methods to investigate cleaning procedures for macro devices to micro devices, with a focus on differences between cleaning at these different scales. The investigations of cleaning procedures with macroscopic methods, namely fluid dynamic

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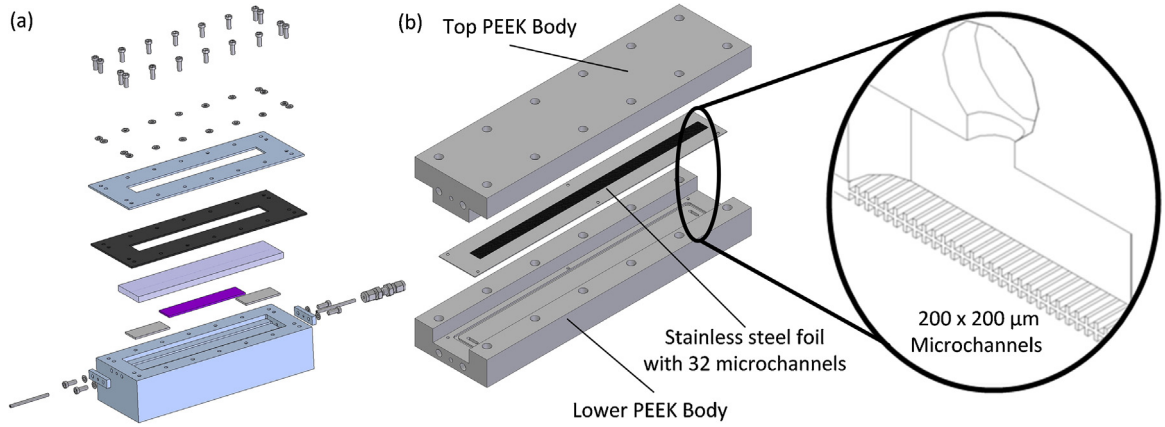

Fig. 1 – (a) Flow channel to evaluate cleaning procedures, 0.2 mm × 0.2 mm × 190 mm. (b) Micro heat exchanger from Karlsruhe Institute of Technology (KIT).

gauging (FDG) and a simple flow channel, are related to the extended *Sinner's circle* (Dürr and Wildbrett, 2006), considering quantity and quality of the soil, substrate and detergent characteristics.

Flow conditions, given by the Reynolds number, within the micro heat exchanger could be mimicked and adjusted easily by the flow channel device for cleaning investigations.

FDG cleaning experiments required a more complex experimental setup compared to the flow channel device. Tuladhar et al. (2000) developed FDG to investigate the thickness of soft fouling layers and as a monitoring tool. The technique also can be used to investigate the shear strength of fouling layers, as shown by Chew et al. for tomato paste [Chew, 2004]. With this technique, a pressure difference between the fluid near the fouling layer surface and a discharge end of the gauge nozzle is set up, establishing flow into the nozzle. Information about the calculation of the resulting shear stress under the nozzle and schematic drawings of FDG devices are given by Tuladhar et al. (2000) and Chew et al. (2004).

2. Experimental

2.1. *Creating fouling layers on stainless steel substrates*

For the investigation of cleaning procedures, standardized LNP fouling layers were created on stainless steel (*AISI 304*) substrates (80 mm × 20 mm × 2 mm). These substrates were put on the bottom of a double walled tank, which was heated to 55 °C, resulting in a substrate surface temperature of 45 °C. After preheating the substrates for 10 min, LNP suspension was sprayed on with a nebulizer (Mark II®-Pharma, WEPA Apothekenbedarf GmbH, Hillscheid, Germany) from a distance of 300 mm. Three spray pulses were applied at 5 min intervals and the water evaporated off between applications. The nebulized suspension mass correlated with the fouling layer thickness, confirmed that controlled and reproducible fouling layer thicknesses could be obtained. This procedure was followed by 24 h of drying before cleaning experiments were carried out. The LNP suspension was prepared with two lipids (carnauba wax (5 wt%), decyl oleate (10 wt%)) and the emulsifier polysorbate 80 (2 wt%) with high pressure homogenization, resulting in a particle diameter of appr. 250 nm and a polydispersity index of ≤0.2, determined by photon correlation spectroscopy. The melting point of the lipid nanoparticles is 80 °C and recrystallization starts at 58 °C.

2.2. *Fluid dynamic gauging*

A detailed description of the apparatus is given by Saikhwan et al. (2006). The fouled substrates were located directly under the gauging nozzle. The resulting wall shear stress τ_w, which affects the fouling layer on the substrates, is calculated from (Chew et al., 2005)

$$\tau_w = \frac{3\eta(\dot{m}/\rho)}{4\pi(h/2)^2(d_t/2)} \quad (1)$$

where η is the dynamic viscosity, $\dot{m}$ the discharge flow rate, ρ the fluid density, h the distance between the discharge nozzle and the fouling layer surface and d_t the inner nozzle diameter (Chew et al., 2005). Within experiments h/d_t was decreased successively until the fouling layer was deformed/pulled off the substrate, determining the shear strength of the fouling layer under the test conditions (fouling layer thickness, temperature, cleaning detergent). Deionized water and RBS®25 (Carl Roth Gmbh & Co. KG, Karlsruhe, Germany) were used as cleaning detergents. Latter is a chemical cleaning detergent, containing emulsifiers.

2.3. *Flow channel*

The second method to investigate a cleaning procedure for the micro heat exchanger was a rectangular flow channel, see Fig. 1(a), with a hydraulic diameter of 800 µm. Fouled stainless steel substrates were studied with varying process conditions, namely cleaning detergent temperature and volumetric flow rate. Higher temperatures of 20–95 °C could be realized than with the FDG apparatus.

2.4. *Continuous crystallization in the micro heat exchanger*

Continuous crystallization experiments were carried out in the micro heat exchanger, see Fig. 1(b). The micro heat exchanger was equipped with a multi-microchannel foil with 32 rectangular microchannels of 0.2 mm × 0.2 mm × 190 mm on both the product and service sides. Detailed description of the process windows of the micro heat exchanger are

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