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High-frequency flow reversal for continuous microfiltration of milk with microsieves

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ABSTRACT

A new filtration method using rotating micro-engineered membranes (microsieves) is described. The method uses constant rotation of the microsieve in combination with high-frequency flow reversal to achieve stable and high fluxes. The high-frequency flow reversal is enabled by a vacuum device placed at the retentate side creating a local negative pressure to purge the microsieve from accumulating particles. The method was validated for milk filtration using microsieves with 0.9 μm circular pores with a trans-membrane pressure (TMP) of 0.05–0.30 bar and flow-reversal frequencies between 5 and 25 Hz. Stable filtration (for at least 4 h) was achieved with permeate fluxes for skimmed milk from 8 to 50 $m^3 h^{-1} m^{-2}$ at room temperature, and for whole milk from 7 to 13 $m^3 h^{-1} m^{-2}$ at 50 °C. The effect of the TMP, rotation frequency and flow-reversal strength on the flux was investigated. The flux increased proportional with the TMP up to a critical TMP of about 250 mbar. Also the flux increased with rotation frequency up to an optimal frequency of 20 Hz. The flow-reversal unit requires a threshold backward flow to create enough negative pressure to overcome the TMP during the removal cycle of the retentate particles. Above this threshold the flux increases strongly with increasing backward flow. The novel high-frequency flow-reversal method enables continuous milk filtration of large volumes during many hours without the need of a chemical cleaning cycle.

1. Introduction

Nowadays, microfiltration is widely spread in the dairy and beverage industry as a method for removal of microorganisms [1–5]. Microfiltration is able to remove bacterial cells and spores in sufficient amounts (3–6 log-reduction), as well as removal of somatic cells without compromising the taste quality and biochemical composition of the milk [1–3,6]. The combination of microfiltration and traditional pasteurization of milk gives raw dairy material with an extended shelf-life, which can later be transformed into products for consumption, such as milk, yoghurt, cheese and protein powder [7–10].

The main challenge in microfiltration, as a pressure-driven process, is membrane fouling, which leads to a significant decline in permeate flux and changes in membrane selectivity over time [11]. Membrane fouling can be reversible or irreversible, and can be further distinguished by extrinsic and intrinsic processes. Extrinsic fouling is caused by particles with a diameter larger than the pore size. These particles cannot enter the pores and accumulate on the surface of the membrane, building-up a concentrated layer of species, also called a cake-layer. Intrinsic fouling is caused by particles with a diameter equal or smaller than the pores, and may adsorb on the inner walls, causing inner pore constriction and inner pore blockage [11,12]. Intrinsic pore blockage increases the inner membrane resistance, while the cake formation creates an additional layer of resistance adding to the whole permeation resistance [13]. Also the retention characteristics of the filtration process itself is not only determined by the membrane layer but also by the permeation characteristics of the cake layer [14].

Membrane fouling in milk microfiltration is usually associated with protein aggregate formation and fat globules. In rare cases precipitation of calcium phosphate at high temperature can result in flux decline as well. The main serum proteins (4–10 nm in size) seem to preferentially adsorb onto the membrane surface upon contact and casein micelles (20–300 nm in size) are introduced into the fouling layer by the application of trans-membrane

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pressure, leading to irreversible permanent changes in surface properties [11,15,16]. Fat globules are an important component of milk as well, and their contribution in the fouling of membranes can be even larger than from proteins. This is actually the main reason why microfiltration of fat-containing dairy products is still not used at industrial scale. The diameter of the fat globules in raw milk varies from 0.1 to 15 μm, with an average 3–4 μm [17]. After homogenization of the milk the average globule size is 0.6 μm and has a narrower size distribution [18]. Casein and serum proteins are thought to help the stabilization of the enlarged surface area of the homogenized fat globules [14,19,20]. From the size of milk proteins and fat globules it follows that proteins mainly cause intrinsic fouling of the microfiltration membrane and that fat globules contribute strongly to extrinsic fouling.

Conventional microfiltration processes are characterized by relatively low permeation rates. This is due to a high ratio between membrane thickness and pore size, an overall low porosity and a complex tortuosity of the membrane channels. Furthermore, the selectivity is low because of a relatively broad pore-size distribution. However, milk microfiltration would ideally be achieved with a high permeation rate at a high selectivity without a reduction in the protein content. This requires membranes with a low flow resistance, i.e. a small membrane inner surface, and a well-controlled pore size distribution of the membrane. For this purpose micro-engineered membranes (microsieves) were introduced into the microfiltration industry [21–23]. Microsieves are characterized by a low flow resistance (owing to a very thin selective silicon-nitride layer), well-defined uniform pores and high pore density. Due to their properties, microsieves demonstrate better separation characteristics at 2 orders of magnitude higher flow rates [22,24]. This makes their application more cost effective and energy efficient, even despite their higher production costs in comparison to conventionally used membranes [13–15,25,26]. Modern manufacturing techniques allow for choosing microsieve pore size, shape, porosity, and surface coatings according to specific needs [27–32]. It is evident that, for example, the pore size of the membrane has to be chosen to be able to prevent permeation of undesirable large particles and allow the passage of desirable smaller ones. Because the operational process flux is 10–100 times larger with microsieves, especially the extrinsic fouling rate is expected to be much higher in comparison to conventional membranes [14,25]. Therefore, effective prevention of cake layer formation on the surface of microsieve is paramount for optimal filtration performance.

Cross-flow is widely used to prevent cake layer formation in filtration processes. Owing to the cross-flow induced shear large particles are effectively dragged away from the surface of the membrane. Required cross-flow velocity to induce sufficient shear to remove particles is dependent on several variables such as the size ratio between the particle and the pore, the trans-membrane pressure and chosen dimensions of the cross-flow channel [14]. But cross-flow is not always sufficient to provide sufficient anti-fouling effect and to maintain a sustainable flux. Therefore, additional pore cleaning measures are necessary. Back pulsing is often applied and in general involves an impulse (for a short time and repeated with a certain time interval) that lifts up the particles accumulated in and on the membrane. The impulse may be caused by a sudden pressure change reversing the flow direction through the membrane. The efficiency of back pulsing as an additional cleaning technique for both conventional membranes and microsieves has been shown in many studies [4,15,16]. The back pulse is essential to inhibit cake layer formation on the top of membrane. High-frequency back pulsing provides effective anti-fouling for a long time, and consequently prolongs filtration time and total throughput [13,15].

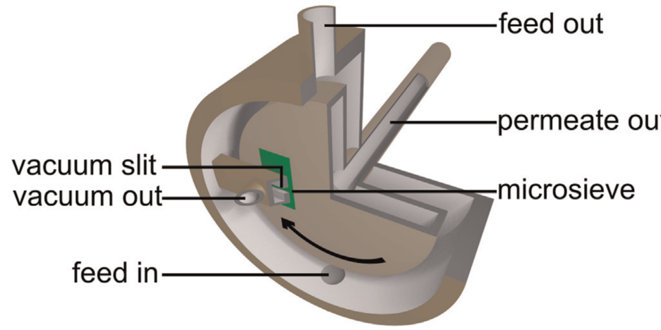

Fig. 1. Schematic image of the rotary microsieve filtration system.

The combination of cross-flow and back-pulsing provides more effective cleaning of the microsieves, improved throughput and separation characteristics [25,33]. This was successfully applied to enable the filtration of milk with microsieves with high fluxes [13,34]. However, the application of microsieve filtration on industrial scale is hampered by the poor scalability of the described back-pulsing methods. The back-pulsing relies on creating a sudden pressure change, either on the feed or the filtrate side of the membrane. This is relatively easy on laboratory scale but to implement this for large volumes at high frequencies is challenging. To overcome this limitation a new system was developed which combines cross-flow and back-pulsing for fouling control with microsieve filtration (Fig. 1) [35]. The microsieves are mounted on a rotating hollow disk inside a filtration vessel chamber through which the feed solution flows. During filtration the disk can be rotated at frequencies between 5 and 25 Hz. The constant rotation of the microsieve creates a shear-effect on the membrane independent of the TMP, as opposed to tubular membrane or other non-rotating membrane systems which suffer from substantial pressure drops along the membrane in order to create sufficient shear [36,37]. The back-pulsing is achieved by reversing the flow through the microsieve with a vacuum device placed close to the surface of the rotating disk with the microsieve. When the vacuum device is operated at high enough flow and is placed in close proximity of the surface, locally a negative pressure drop is created. The microsieve will pass the vacuum device every rotation and the pressure drop will reverse the flow through the microsieve. Consequently, the flow-reversal frequency is directly related to the shear stress at the microsieve surface. Due to the rotation frequency of 5–25 Hz, high-frequency flow reversal is achieved over a very small defined area of the microsieve ($<1\%$ of total area) in order to minimize filtrate loss.

In this study the performance of the novel rotating microsieve filtration device with high-frequency flow reversal is evaluated for microfiltration of skimmed and full-fat milk. The effect of trans-membrane pressure, rotation frequency and flow-reversal strength on the flux is determined. The systems efficiency for fouling control is shown by filtration of full-fat milk. Furthermore, long-term experiments were conducted to demonstrate the potential for applying the system for prolonged filtration of milk.

2. Materials and methods

2.1. Materials

Commercial pasteurized and homogenized skimmed and full-fat milk (0.0% fat and 3.6%, respectively) were obtained from FrieslandCampina and stored at 4–6 °C. During the experiments skimmed milk was kept in an open reservoir placed in an ice bath to maintain the temperature at 20 ± 2 °C. The ice cooling of the milk feed was needed to avoid the heating up of the milk during the experiments. The main source of heat was the diaphragm