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Effect of pore size and process temperature on flux, microbial reduction and fouling mechanisms during sweet whey cross-flow microfiltration by ceramic membranes

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ABSTRACT

In this study, operating conditions for the cross flow microfiltration (CFMF) of sweet whey were optimised. Filtrations were performed for 65 min at 20, 40 and 50 °C using ceramic membranes with different nominal pore sizes (0.1, 0.5 and 0.8 μm). Periodically, samples of whey retentate and permeate were taken and analysed for microbiological quality and physical and chemical properties. The best microbial reduction rates were achieved during filtration using a 0.1 μm membrane at 50 °C. The highest flux rates were achieved during filtration at 50 °C with all tested membranes. Fouling intensity was the lowest after filtration using a 0.5 μm membrane at 20 °C. According to all results obtained, the membrane with the nominal pore size of 0.5 μm appeared to be optimal for purposes of preserving the nutritional value, minimising membrane fouling and achieving appropriate microbiological quality of sweet whey.

1. Introduction

Whey has excellent nutritional value, mostly due to whey proteins. It has been proven that whey proteins, especially α-lactalbumin (α-La) and β-lactoglobulin (β-Lg), are a source of bioactive peptides with several health effects, such as antimicrobial, antihypertensive and anticarcinogenic properties (Chatterton, Smithers, Roupas, & Brodkorb, 2006). Additionally, whey proteins have excellent functional properties and are widely used in the food industry (Foegeding, Davis, Doucet, & McGuffey, 2002). Since fresh whey is very susceptible to microbial spoilage, heat treatments like pasteurisation are obligatory. Whey proteins tend to denaturation and precipitate at temperatures above 60 °C (Parris, Purcell, & Ptashkin, 1991), so sediments of proteins and some salts are likely to occur during heat treatment (Jeličić, Božanić, & Tratnik, 2008), causing a reduction in the nutritional and sensory quality of whey. Also, as a result of denaturation, the potential to further modify functional properties of whey proteins is often lost (Kulozik & Kersten, 2002).

The world whey production is over 160 million tons per year, with an annual growth rate of 1–2% (Guimarães, Teixeira, & Domingues, 2010). About 70% of total whey is processed into different products, but about 30% of whey is being utilised for pig feeding or other similar purposes (Jelen, 2003). To increase whey utilisation for human nutrition, alternative processing techniques such as membrane processes, i.e., microfiltration (MF) or ultrafiltration, are needed.

MF is commonly used to remove the microorganisms present, as well as their thermo stable spores, fat globules and other lipid components (e.g., lipoproteins), and casein residues (Goulas & Grandison, 2008). Kaufmann and Kulozik (2006) pointed out that, regardless of the processing parameters, an average microbial reduction of vegetative cells from approximately 3–4 log cycles can be observed during MF of skimmed milk.

The high variability of whey composition, the high water content and the high numbers of lactic acid bacteria make microfiltration of fresh whey slightly more complicated than milk, in terms of membrane fouling. Calcium phosphate salts, as well as whey proteins, naturally tend to precipitate at higher temperatures (e.g., 50 °C), and are considered to be the main contributors to membrane fouling (Goulas & Grandison, 2008; Kelly & Zydney, 1997; Koutsoukos, 2007). In recent years, a lot of efforts have been made to improve MF technology. However, the main

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disadvantage characterising most of the proposed methods is that they cannot be applied in a large scale. In addition to that, some also negatively affect the quality of the filtered product (Brans, Schroën, van der Sman, & Boom, 2004).

Only a few comprehensive studies have been focused on optimising the efficiency of sweet whey cross flow microfiltration (CFMF) in terms of the microbial reduction, the whey protein recovery, and membrane fouling. Hence, the aim of this study was to investigate CFMF of sweet whey by using ceramic membranes with different nominal pore size in relation to the process temperature. Microbial reduction, flux, membrane fouling parameters, whey protein retention and calcium permeation were monitored. Also, to compare CFMF as a possible alternative for purposes of microbiological stabilisation of whey, microbiological reduction and whey protein recovery have been compared with that obtained by the conventional pasteurisation.

2. Materials and methods

2.1. Whey samples

To be able to use whey samples of same characteristics and composition in all filtrations, a process of whey production with high reproducibility needed to be established first.

Whey was produced from skimmed and pasteurised (71 °C for 20 s) milk obtained from the dairy industry Molkerei Weihenstephan GmbH & Co. KG, Freising, Germany. Approximately 40 L of pasteurised skim milk was first inoculated with previously prepared mesophilic culture F-DVS Flora Danica (Chr. Hansen, Hørsholm, Denmark) and incubated until reaching pH value of 6.15. Subsequently, 2% (v/v) of liquid rennet with coagulating power of 1:15,000 (Chr. Hansen) was added, followed by incubation at 35 °C for 20–30 min. The curd obtained was cut, periodically stirred and heated until the maximum whey amount (~25 L) was separated. Samples of the produced whey were analysed for the total protein content (Dumas method; AOAC, 1995), the content of α-La, β-LgB and β-LgA [reversed phase-high performance liquid chromatography (RP-HPLC) method; Kessler & Beyer, 1991]. Also, the calcium content (flame photometry), pH value, viscosity (capillary viscometer) and density (digital densitometer) were determined. All of the methods are described in Section 2.3. The average whey composition is presented in Table 1.

2.2. Filtration procedure and calculations

2.2.1. General procedure and filtration rig

The filtrations were performed with approximately 20 L feed that was recirculated for 65 min at 20, 40 or 50 °C. Multichannel α-alumina tubular ceramic ZrO_2, membranes (length of 1.020 m, internal diameter of 6 mm, Membralox series, Pall GmbH, Dreieich, Germany) with nominal pore sizes of 0.1, 0.5 and 0.8 μm were used. The effective filtration area was 0.24 m^2. The laboratory filtration rig was the same as that previously described by Piry et al. (2008), with the exception that the membrane was not subdivided into sections.

The whey and the microfiltration (MF) unit were equilibrated at the required temperature prior to each experiment. The measurements related to water flux and membrane fouling resistance calculations were performed as described in Piry et al. (2008). The sampling of whey retentate and permeate was performed every 5 min during the first 15 min of filtration, and afterwards every 10 min. In the same manner, permeate flux (J) was measured gravimetrically. All samples were analysed for the content of α-La, β-LgB and β-LgA, as described in Section 2.3, and for total bacterial count and the coliform bacteria count, as described in Section 2.4.

After a filtration experiment with whey, the plant was flushed with demineralised water and then cleaned in a three-step procedure, as described in Piry et al. (2008). The cleaning efficiency was controlled by measuring water flux at standard conditions (64% working capacity of the pump, $T = 55$ °C, $\Delta p \sim 2 \times 10^5$ Pa).

2.2.2. Filtration parameters

For all filtrations the centrifugal pump was adjusted to 64% working capacity, which corresponded to an average transmembrane pressure (TMP) between $1.25 \pm 0.02 \times 10^5$ Pa and $1.64 \pm 0.02 \times 10^5$ Pa, depending on the process temperature and the used membrane. This corresponds to a retentate flow velocity of approximately 6 m s^{-1}, which is typical for industrial scale filtration.

The total filtration resistance R_T was calculated according to Darcy's law (Eq. (1)):

$$R_T = \mathrm{TMP} \Big/ \left(\eta_p \times J_p\right) \quad (1)$$

with η_p representing permeate viscosity and J_p representing permeate flux.

The membrane resistance R_M was calculated using a linear regression method as Konieczny and Klomfas (2002) described in detail according to Eq. (2).

$$J = \mathrm{TMP}/(\eta \times R_M) \quad (2)$$

with J representing water flux and η representing water viscosity.

For water a viscosity of 5.47×10^{-4} Pa s (50 °C), 6.53×10^{-4} Pa s (40 °C), 10.02×10^{-4} Pa s (20 °C) respectively, were used for the calculations. For MF permeate average viscosities were measured as follows: 6.48×10^{-4} Pa s at 50 °C, 7.29×10^{-4} Pa s at 40 °C and 11.9×10^{-4} Pa s at 20 °C. During the filtration of whey, reversible fouling resistance (R_{rev}) and irreversible fouling resistance (R_{irev}) were calculated by Eqs. (3) and (4) (Heino, 2009):

$$R_T = R_m + R_{irev} + R_{rev} \quad (3)$$

$$J_{H_2O} = \mathrm{TMP}/(\eta_{H_2O} \times R_{irev}) \quad (4)$$

with J_{H_2O} representing water flux after flushing with demineralised water at the end of filtration, before cleaning the membrane with chemicals as described in Piry et al. (2008).

Table 1
Composition and properties of rennet cheese whey.[a]

Parameter	Average
pH	6.15 ± 0.02
α-Lactalbumin (g L^{-1})	0.825 ± 0.032
β-Lactoglobulin B (g L^{-1})	1.140 ± 0.110
β-Lactoglobulin A (g L^{-1})	2.660 ± 0.163
True protein (%)	0.493 ± 0.091
Dynamic viscosity (kg m^{-1} s^{-1}) × 10^{-3}	
at 20 °C	1.231 ± 0.09
At 40 °C	0.829 ± 0.20
At 50 °C	0.729 ± 0.13
Density (kg m^{-3})	1.0248 ± 0.018
Ca (mg L^{-1})	448.49 ± 41.57

[a] Values are the average of triplicate analyses ± standard deviation.