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Separation and recovery of proteins and sugars from Halloumi cheese whey

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ABSTRACT

Halloumi is the traditional cheese of Cyprus that is produced using typically two processes: milk coagulation and pressed curd cooking. Both processes generate two whey substrates rich in proteins and sugars. The scope of the current study is to investigate the separation and recovery of these compounds by processing samples with a cross-flow ultrafiltration module and five membranes in two sequential steps. Three membranes (100, 50 and 20 kDa) were tested in the first scenario and 20 kDa-permeates were assayed in the second using the two materials with narrower pores of 2 and 1 kDa. Experiments were conducted under constant temperature, circulation flux and several transmembrane pressures, while recovery monitoring was performed by determining operation parameters and retention of proteins, reducing and non-reducing sugars. Results indicated that ultrafiltration is able to separate the target compounds optimally upon two different approaches: using a 100 kDa-polysulphone barrier or by combining 20 kDa-polysulphone and 2 kDa-polyethersulphone membranes. The recovery of proteins in the first approach was high for both samples (69–76%), while the retention of non-reducing sugars (herein expressed mainly lactose) was negligible (2–7%). Using the combined treatment, the recovery of proteins and non-reducing sugars was almost quantitative (87–90%) and rather low (39–32%), respectively.

1. Introduction

Recovery of value-added compounds from food by-products has been proposed to progress in five distinct stages: (i) macroscopic pretreatment, (ii) separation of macro- and micro-molecules, (iii) extraction, (iv) purification and (v) product formation (Galanakis, 2013). Membrane technologies like micro- (MF), ultra- (UF) and nano-filtration (NF) have been successfully used in the corresponding first, second and fourth stages, as they are able to remove suspended solids, concentrate macromolecules and clarify them from smaller components, respectively. Membranes have also been applied to pre-concentrate milk, separate cells and lactic acid from fermentation broths, standardize protein and fat ratios prior to conventional cheese-making, and finally valorize whey (Almecija, Ibanez, Guadix, & Guadix, 2007; Gonzalez, Alvarez, Riera, & Alvareza, 2008). The latter is the most investigated food by-product for recovery purposes (Galanakis, 2012). Indeed, whey comprises one of the biggest reservoirs of food proteins and sugars (mainly lactose) and are currently separated with sequential membrane processes. The latest compounds are typically separated simultaneously with two membranes in respective sequential processes. In the first stage, a UF membrane possessing wide pores in the range of 300 to 10 kDa is applied in order to separate globular proteins from sugars (Almecija et al., 2007; Baldasso, Barros, & Tessaro, 2011; Cowan & Ritchie, 2007; Marella, Muthukumarappan, & Metzger, 2011). In the second stage, a UF or NF membrane with narrow pores (<10 kDa) is used to recover lactose in the permeate stream (Atra, Vatai, Bekassy-Molnar, & Balint, 2005; Cuertas-Uribe et al., 2009; De Souza et al., 2010). Other investigators have suggested a two-step tangential flow UF system (100 and 30 kDa) in order to fractionate sequentially bovine serum albumin, β-lactoglobulin, and α-lactalbumin (Cheang & Zydney, 2004). In any case, process optimization should be carried out for each whey type separately, since:

(a) whey is a multi-component suspension and variations of feed content may cause fouling phenomena (Macedo, Duarte, & Pinho, 2011).
(b) Protein and sugar ratio is critical in the first stage, as macromolecules may block membrane pores and this way increasing the retention of smaller compounds.
(c) Lactose fermentation and hydrolysis degree is important in the second stage since its molecular weight is two- and four-fold higher than lactic acid and galactose (or glucose), respectively.
(d) Heat treatment and protein denaturation affect the physicochemical properties during processing.

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Halloumi is the traditional cheese of Cyprus that is produced using mixtures of ovine and caprine milk (Kaminarides, Stamou, Massouras, & Georgala, 2009; Papademas & Robinson, 2002). The distinctive production (Fig. 1) includes: (a) heating of milk at 35–36 °C (40–60 min) with or without the presence of a starting culture, (b) cooking of pressed curd blocks in whey for 0.5–1 h at 95 °C and (c) storage in the brine for 1–2 days. During heat treatment, proteins begin to coagulate at ~70 °C and respective rate increases at 90 °C. During storage of Halloumi cheese in the brine, an acceleration of undesirable metabolites (lactic acid or, ethanol) in the corresponding whey may be observed (Kaminarides, Stamou, & Massouras, 2007).

The recovery of proteins and sugars from Halloumi whey has not been investigated yet, while studies concerning the simultaneous separation of both valuable compounds from similar whey types are relatively limited. Herein, two fresh whey samples obtained prior to and after the cooking process of Halloumi manufacture were processed in two sequential UF steps with different membranes. Experiments were carried out under different transmembrane pressures (TMP:s) and monitoring was performed by determining permeate flows and retention coefficients of proteins and sugars.

2. Experimental

2.1. Materials

Reagents were of analytical grade. Fresh samples of Halloumi whey (A and B) were collected from a local dairy industry (Lemesos, Cyprus) as illustrated in Fig. 1. Halloumi cheese was prepared using a mixture of ovine and caprine milk. Samples were kept in plastic containers in the freezer (−20 °C) until usage. Sample B was pre-filtered with a cloth prior to utilization as a feed liquid in order to avoid membrane blocking from protein precipitation in acidic environment. Sample A was directly applied. Five commercial ultrafiltration membranes (Alfa Laval, Nakskov, Denmark) were studied in the current investigation possessing a molecular weight cut-off (MWCO) from 100 to 1 kDa (Table 1): GR40PP, GR51PP, GR70PP (polysulphone), GR95PP (polyethersulphone) and ETNA01PP (composite fluoro polymer).

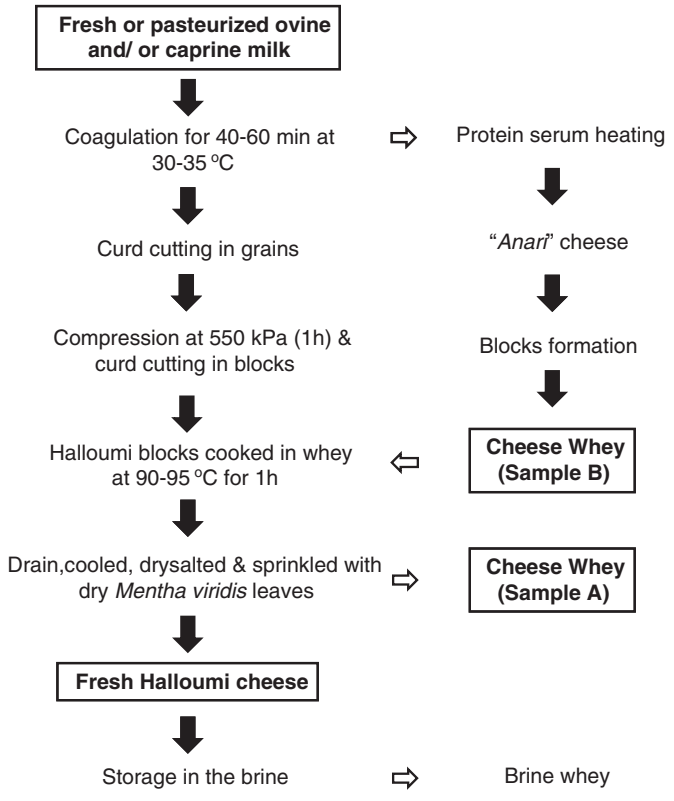

Fig. 1. Diagram of Halloumi cheese production and generation of respective whey samples.

2.2. Experimental set-up and operation control

Membrane experiments were carried out with a cross-flow UF module (DSS Labstak M10, Alfa Laval Nakskov, Denmark) using the set-up described by Galanakis, 2013. The total membrane area was 0.1344 m^2 (4 membrane sheets of 0.0336 m^2) and the processed feed volume was 3 L, while the recycling flow rate and processing temperature remained constant at 85 $mL \cdot s^{-1}$ and 25 ± 0.5 °C, respectively. Initial permeate flux was measured gravimetrically as the variable of permeate weight with time and expressed in $L\ m^{-2}\ h^{-1}$.

2.3. Experimental procedure

2.3.1. Membrane pretreatment

Each of the five membrane types was placed into the cross-flow module and pre-treated with de-ionized water as feed liquid (3 L) in order to avoid membrane compaction during UF experiments and wash out preservatives such as glycerine (Galanakis, Fountoulis and Gekas, 2012). GR40PP was pressurized at 1, 2, 3, 2 and 1 bar in two sequential rounds (15 min duration). GR51PP and GR70PP were pressurized between 1 and 4 bar, while GR95PP and ETNA01PP were pressurized in the range of 1–5 bar (1 bar ~ 100 kPa) as above.

2.3.2. Separation experiments

Pretreated membranes were sequentially utilized in separation experiments for each feed–membrane combination. Three liters of feed liquid (samples A and B, and corresponding 20 kDa-permeates) was processed in the membrane apparatus and pressurized in the same TMP:s as above (Table 1) in two sequential rounds (15 min duration). Permeate initial flux was determined during the process and the relative flux (RF) of the feed solutions was calculated in percentage according to the following equation (Patsioura, Galanakis, & Gekas, 2011):

$$RF = \frac{J_v}{J_{w0}} \cdot 100(\%) \quad (1)$$

where J_v is the permeate initial flux and J_{w0} is the pure water initial flux. The initial permeability of water (L_w) or feed (L_v) was quantified by the following equations (Galanakis, Markouli, et al., 2013):

$$L_w = \frac{J_{w0}}{TMP} \quad (2)$$

and

$$L_v = \frac{J_v}{TMP}. \quad (3)$$

The intrinsic membrane resistance of water (R_{mw}) or feed (R_{mv}) (expressed in Terra m^{-1}) was calculated by the following equations:

$$R_{mw} = \frac{TMP}{\mu_w \cdot J_{w0}} \quad (4)$$

and

$$R_{mv} = \frac{TMP}{\mu_v \cdot J_{v0}} \quad (5)$$

respectively, where μ_w and μ_v are the viscosities of pure water and feed solution, respectively.

After the total 15 minute pressurized time at the highest applied TMP, UF module was operated for 60 minute prior to collecting samples