Innovative Food Science and Emerging Technologies 21 (2014) 131–141

Contents lists available at ScienceDirect

Innovative Food Science and Emerging Technologies

journal homepage: www.elsevier.com/locate/ifset

Combination of homogenization and cross-flow microfiltration to remove microorganisms from industrial buttermilks with an efficient permeation of proteins and lipids

Jacques Fauquant, Eric Beaucher, Carine Sinet, Benoit Robert, Christelle Lopez *

INRA, UMR 1253 STLO, 65 rue de Saint-Brieuc, F-35000 Rennes, France
Agrocampus Ouest, UMR 1253 STLO, 65 rue de Saint-Brieuc, F-35000 Rennes, France

ARTICLE INFO

Article history:
Received 12 July 2013
Accepted 7 October 2013

Editor Proof Receive Date 7 November 2013

Keywords:
Buttermilk
Microstructure
Polar lipid
Milk fat globule membrane
Bacteria

ABSTRACT

Buttermilk is a source of interesting nutritional and functional components, e.g. polar lipids and proteins. However, it is still considered as a low-value by-product of the dairy industry with high variations in biochemical composition and bacterial contaminations. The objective of this study was to develop a process based on microfiltration, permitting the removal of microorganisms to ensure the safety of buttermilk components for human nutrition. Industrial buttermilks and the products collected during microfiltration were characterized using particle size measurements, biochemical and microbiological analysis. The combination of homogenization at 80 MPa and cross-flow microfiltration successfully removed bacteria from skimmed buttermilk: bacterial reduction > 4.8 $\log_{10}$ with 0 cfu/ml in the permeate using the 0.8 μm pore size membrane and 1 cfu/ml with 1.4 μm membrane. Chemical analysis revealed the efficient permeation of proteins, total lipids and polar lipids. Polar lipid classes permeated equally the membrane. This work will contribute in improving the safety of buttermilk-based ingredients.

Industrial relevance: This work describes the development of an innovative process combining homogenization and cross-flow microfiltration for the selective removal of bacteria from industrial buttermilks. This process is an alternative to heat treatments that alter the nutritional and organoleptic properties of food products. The safety of buttermilk-based ingredients containing milk polar lipids of interest will contribute in their economic valorization for human nutrition.

1. Introduction

Buttermilk is a by-product of the dairy industry obtained during the manufacture of butter, which is chemically considered as skim milk enriched with milk fat globule membrane (MFGM) material. Industrial buttermilks are considered as low-value products with high variations in their biochemical composition, bacterial contaminations, and with limited functional properties. Hence, buttermilks are dried into powders and mainly given for animal feeding. Only a limited amount of buttermilks is used in the food industry as ingredients for human consumption. However, buttermilk is a source of interesting components from a functional and nutritional point of views, e.g. polar lipids from the MFGM, proteins (caseins, whey proteins, membrane proteins), lactose and minerals. The added value of buttermilk is currently minimal and its full potential still remains to be exploited by the development of new technologies. In this study, we focused on the development of a process permitting the removal of microorganisms to ensure the safety of buttermilk components, e.g. for human nutrition.

During the manufacture of butter, materials derived from the MFGM are recovered in the aqueous phase called buttermilk when milk fat globules are mechanically disrupted upon churning of cream (Keogh, 2006). MFGM fragments contain polar lipids (glycerophospholipids and sphingolipids), neutral lipids (mainly high melting point saturated triacylglycerols), proteins, glycoproteins (e.g. butyrophilin, mucins), enzymes (e.g. xanthine oxidase/dehydrogenase), cholesterol and other minor compounds (Dewettinck et al., 2008; Lopez, 2011). The MFGM is considered to be a rich source of bioactive lipids and membrane proteins (Spitsberg, 2005; Vesper et al., 1999). More information becomes available on the health benefits associated with the consumption of MFGM components (see reviews Dewettinck et al., 2008; Lopez, 2011). Particularly, milk phospholipids and sphingolipids have been reported to have interesting biological, nutritional and health properties (Vesper et al., 1999; review Lopez, 2011). Buttermilk components also have interesting functional properties, such as foaming and emulsifying properties (Corredig & Dalgleish, 1998), that are used in several food applications (e.g. bakery, ice-cream manufacture, chocolate, low-fat products). The production of buttermilk fractions enriched in polar lipids can potentially be used as functional and health ingredients in human nutrition (Corredig, Roesch, & Dalgleish, 2003; Ward, German, & Corredig, 2006). These products will extend marketing opportunities

* Corresponding author at: INRA, UMR 1253 STLO, 65 rue de Saint-Brieuc, F-35000 Rennes, France. Tel.: +33 2 23 48 56 17; fax: +33 2 23 48 53 50.
E-mail address: Christelle.Lopez@rennes.inra.fr (C. Lopez).

1466-8564/$ – see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.ifset.2013.10.004

by giving the food industry ingredients that fulfill specific needs with a great economic impact.

For the last 15 years, industrials and academic research teams developed processes able to selectively fractionate buttermilk components, and particularly to recover and concentrate MFGM fragments and milk polar lipids from buttermilks and from other products such as butter serum and cheese whey. The different steps involved in the recovery of MFGM materials have been reviewed (Dewettinck et al., 2008; Ward et al., 2006). However, no studies focused on the selective removal of microorganisms from buttermilks in order to assure their safety. This point is of utmost importance considering the use of buttermilk and buttermilk fractions as an ingredient for food applications in human nutrition.

Buttermilk is a nutritious medium that represents a favorable environment for the growth of spoilage and pathogenic microorganisms for human beings (e.g. *Listeria*, *Brucella*, *Mycobacterium* or *Salmonella*). Hence, industrial buttermilks contain a microbial flora formed by numerous species resulting from the potential contaminations occurring during the manufacture of butter and/or due to transportation equipment and storage conditions of buttermilk before drying. Techniques have been reported to be efficient to destroy microorganisms, such as ultrasounds and ultra-high pressure homogenization (≥100 MPa). However, destruction of the microorganisms has been mostly done for sixty years by applying to fluids heat treatments such as pasteurization or sterilization performed at ultra-high temperature. Heat treatments lead to the thermal denaturation of milk proteins and to the formation of heat-induced compounds (e.g. chemical components resulting from Maillard reaction), and then alter the nutritional and organoleptic properties of the product. Heat treatments also negatively affect the ability of milk components to be transformed in cheeses (problems of coagulation). Separation technologies based on the use of membranes, such as microfiltration, offer an interesting alternative to heat treatments. Cross-flow microfiltration is used in the dairy industry as an industrial membrane separation technology for the removal of microorganisms from skimmed milk (Holm, Malmerg, & Svensson, 1986; Huffman & Harper, 1999; Saboya & Maubois, 2000; Schmidt, Kaufmann, Kulozik, Scherer, & Wenning, 2012). For this application, microfiltration membranes with an average pore size of 1.4 μm are generally used, leading to a decrease of the bacterial counts by 4 to 6 $\log_{10}$ units (Schmidt et al., 2012). The retention of microorganisms with various cellular volumes (i.e. from 0.7 to 4.1 μm^3) and various morphologies found in milk-originated fluids has been investigated with 1.4 μm membranes (Trouvé et al., 1991). The debacterized milk produced with 1.4 μm membrane is transformed into consumption fluid milks (e.g. Marguerite® milk in France, Purfiltre® milk in Canada), cheeses or long storage dairy products such as low-heat milk powder or protein derivates (Saboya & Maubois, 2000). The use of microfiltration with 1.4 μm membrane can be assimilated to a cold pasteurization (Trouvé et al., 1991). Using membranes with 0.8 μm pore size to microfiltrate skimmed milk, Lindquist reported a bacterial removal increased by 2 to 3 $\log_{10}$ compared to the 1.4 μm pore size membrane (Lindquist, 1998). Hence, decreasing the mean pore size of the filtration membrane could increase the efficiency of bacteria removal. The removal of microorganisms from lipid-containing dairy fluids is much more challenging, since lipids can contribute in the fouling of the membranes during microfiltration. This is the main reason why microfiltration of lipid-containing dairy fluids is not currently used at the industrial scale. Regarding particularly buttermilks, no studies or processes involving cross-flow microfiltration for their microbial removal have been reported in literature.

The objectives of this study were to define a technological process able to remove microorganisms from industrial sweet cream buttermilks by using cross-flow microfiltration with 0.8 and 1.4 μm pore size membranes. For this purpose, we characterized the microstructure and size distribution of particles in industrial sweet cream buttermilks and investigated the effect of homogenization on the efficiency of microfiltration. The biochemical composition of the permeates and retentates recovered during microfiltration have been characterized.

2. Materials and methods

2.1. Industrial buttermilks

For each trial, 1000 L of buttermilk were provided by Coopérative Agrilait (Cesson Sévigné, France). Buttermilks were produced at the industrial scale during the manufacture of butter using a continuous butter churn from sweet cream (n = 9 buttermilks). Only fresh liquid sweet cream buttermilks were used in this study.

2.2. Processing

Several technological steps have been used to develop the process. Centrifugation of buttermilks was performed at 40 °C using a cream separator (Elecrem, Westfalia, Fresnes, France). Homogenization of buttermilks was performed using a Rannie Lab 12/51H two-stage homogenizer (ATS, Moissy Cramayel, France). The buttermilks were heated to 45 ± 1 °C before homogenization. On the first stage of the homogenizer, various pressures ranging from 5 to 80 MPa have been applied. On the second homogenizing valve, we applied 10% of the first-stage pressure. Cross-flow microfiltration was performed with a pilot rig (TIA, Bollène, France) equipped with 0.24 m^2 of tubular mineral multichannel membrane made of alumina (19 channels of 4 mm diameter; 1P19-40; Sterilox®, Pall Exekia, Tarbes, France). We used membranes with an average nominal pore size of 0.8 μm and 1.4 μm (Sterilox® membranes). Buttermilk was heated continuously at 57 °C with a plate exchanger and driven by a feed pump. A centrifuge pump (Fristam, Noisy-le-Sec, France) was used for retentate recirculation. Another centrifuge pump (Fristam S.A.) allowed a cocurrent permeate recirculation allowing the hydraulic concept of low and uniform transmembrane pressure all along the membrane. The pilot rig was washed and sterilized before each experiment. For microfiltration with the 0.8 μm and 1.4 μm pore size membranes, the hydrodynamic parameters were as follows: (i) Temperature: T = 57 ± 1 °C, (ii) feed pressure = 180 kPa, (ii) volume reduction factor, VRF = 20 ± 1, (iii) Permeation flux Jp = 200 L/h/m^2 (for 0.8 μm) and Jp = 300 L/h/m^2 (for 1.4 μm), (iv) Fluid velocity = 7 m/s (given by the recirculation pump); flow = 6 m^3/h. Transmembrane pressure (TMP, kPa) and permeation flux (Jp, L/h/m^2) were determined as a function of time during the microfiltration process. Permeates and the corresponding retentates have been collected for biochemical, physicochemical and microbiological analysis. The products have been lyophilized and stored at −20 °C until further biochemical experiments.

Transmission rate (%) of proteins, total lipids and polar lipids through the membrane was calculated according to Equation (1): $Tr\ (\%) = (C_{permeate}/C_{retentate}) \times 100$, where $C_{permeate}$ is the concentration of a component in the permeate side and $C_{retentate}$ is the concentration of the same component in the retentate side. Recovery yield (%) of proteins, total lipids and polar lipids through the membrane was calculated according to Equation (2): Recovery yield (%) = ($C_{permeate}/C_{feed}$) × 100, where $C_{permeate}$ is the concentration of a component in the permeate side and C_{feed} is the concentration of the same component in the product before microfiltration. Rejection, that indicates bacteria removal by membrane and then measure the efficiency of microfiltration, was calculated according to Equation (3): Rejection (%) = $(1 - C_{permeate}/C_{feed}) \times 100$, where $C_{permeate}$ is the concentration of bacteria in the permeate and C_{feed} is the concentration of bacteria in the product before microfiltration.

2.3. Particle size measurements

The particles size distributions in the buttermilks, and in the products recovered during microfiltration (retentates and permeates)