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The lipid content and microstructure of industrial whole buttermilk and butter serum affect the efficiency of skimming

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ABSTRACT

The processes developed to valorize buttermilks and butter serums generally start by a technological step aiming at removing lipids by centrifugation. The efficiency of this skimming step has never been studied yet. The objective of this study was then to characterize the efficiency of the skimming of industrial buttermilk and butter serum and to determine its consequences on the lipid composition and microstructure of related products. This work clearly shows that the efficiency of the skimming step, operated both at pilot and industrial scales, is never complete and depends on the characteristics of the fluids to be treated. There exists a threshold of lipid content (7% total lipids in dry matter for buttermilk; 20% for butter serum) under which the skimming is not efficient. Above this threshold of lipid content, the skimming step removes all particles with a size larger than 1 μm (large fat globules, butter fines), but does not succeed in removing small size lipid fraction (fragments of membrane, lipid vesicles), which results in an increase in the polar lipids on total lipid ratio for both products (up to 20 ± 5% and 49 ± 6% for buttermilks and butter serums respectively). The difference of skimming efficiency, threshold values of skimming, and composition of final products between the buttermilks and butter serums could be attributed to differences in the microstructure of the lipids. Even if not totally efficient, the skimming step leads to a standardization of total lipid content of buttermilks and butter serums, useful for a better control of technological processes aiming at valorizing individual compounds of these dairy by-products.

1. Introduction

Buttermilk and butter serum are both by-products of the milk fat industry. Buttermilk is the liquid phase released during cream churning in the process of butter manufacture. Butter serum is the aqueous phase obtained after melting and centrifugation of butter in the production process of anhydrous milk fat (AMF). AMF consists of a 99.9% pure milk fat product with specific techno-functional properties like high resistance to heat and mechanical treatments (Boutonnier, 2008; Vanderghem et al., 2010). The large production of butter and AMF worldwide (10 Mt of butter and AMF in 2013, Cniel, 2015) results in a large production of buttermilks and butter serums which are mainly mixed together (with a higher proportion of buttermilk due to a higher production of butter) and spray-dried into powder for animal feed.

In terms of composition, buttermilk and butter serum are very close to skimmed milk when focusing on proteins, ashes and lactose content (Corredig & Dalgleish, 1997; Vanderghem et al., 2010). However, they largely differ in terms of lipid fractions. Buttermilk and butter serum contain respectively about 4.6–14.5% and 24% fat in dry matter (Vanderghem et al., 2010). They offer high concentrations of functional polar lipids in comparison with skimmed milk, whole milk and cream (Christie, Noble, & Davies, 1987; Mcpherson & Kitchen, 1981; Rombaut, Van Camp, & Dewettinck, 2006; Vanderghem et al., 2010), with concentration values reaching 1.2–2.1 and 11.5% polar lipids on dry matter for buttermilk and butter serum respectively (Vanderghem et al., 2010). The polar lipids of these by-products are mainly composed of phospholipids (phosphatidylcholine, PC; phosphatidylethanolamine, PE; phosphatidylserine, PS; and phosphatidylinositol, PI), and sphingolipids (mainly sphingomyelin, SM) with a higher sphingomyelin proportion in polar lipids in butter serum than in buttermilk (Britten, Lamothe, & Robitaille, 2008; Rombaut & Dewettinck, 2006; Rombaut et al., 2006). The polar lipids are initially present in the biological milk fat globule membrane (MFGM), within proteins, cholesterol and neutral lipids (Danthine, Blecker, Paquot, Innocente, & Deroanne, 2000; Lopez, 2011; Singh, 2006) and are released in the aqueous phases during cream churning. During this process, the milk fat globules present in the cream are actually destabilized by mechanical agitation in the presence of air, which results in the breakage of MFGM, in the reversal of oil-in-water emulsion into a water-in-oil emulsion and in a flow of polar lipids

Abbreviations: AMF, anhydrous milk fat; CLSM, confocal laser scanning microscopy; DM, dry matter; HPLC, high-performance liquid chromatography; MFGM, milk fat globule membrane; PC, phosphatidylcholine; PE, phosphatidylethanolamine; PI, phosphatidylinositol; PS, phosphatidylserine; TAG, triacylglycerol; TEM, transmission electron microscopy; SM, sphingomyelin.

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towards the buttermilk phase (Rombaut et al., 2006). In terms of lipid structure, the polar lipids are present in buttermilk in the form of MFGM fragments (Rombaut et al., 2006) with highly variable sizes as observed by microscopy (Morin, Jiménez-Flores, & Pouliot, 2007) and/or in the form of polar lipid vesicles as suggested by Fauquant, Beaucher, Sinet, Robert, and Lopez (2014). In the industrial butter serums, lipids are organized in the form of MFGM fragments and vesicles (Lopez, Cauty, & Guyomarc'h, 2015).

Due to their interesting lipid composition, efforts have been made for more than 20 years by industrial and academic research teams, to develop new valorization processes of buttermilk and butter serum and to produce nutritional and functional high added-value ingredients (Dewettinck et al., 2008; Gassi et al., 2015; Vanderghem et al., 2010). Regardless of the process, buttermilk and butter serum are systematically skimmed, right from the very start of the process. The skimming is carried out for two main reasons. It makes possible the recovery of some residual lipid (such as butter fines or milk fat globules, like in the skimming of milk and cheese wheys) (Fauquant et al., 2014; Morin et al., 2007) that can be valorized in another process. It reduces the residual lipid content of final ingredients which hampers the spray-drying process (Boutonnier, 2008). However, despite the systematic use of skimming, the effect of this processing step on lipid composition and structure of treated buttermilk and butter serum has never been investigated and discussed in the literature. Fauquant et al. (2014) and Morin et al. (2007) were the only authors reporting a specific study on buttermilk skimming performances. According to Fauquant et al. (2014), skimming of industrial buttermilks decreased the total lipid content by about 0.5 to 1 $g \cdot kg^{-1}$. Morin et al. (2007) mainly focused on the impact of pasteurization of cream prior to churning on lipid removal. They reported that the pasteurization of cream leads to difficulties in the separation of lipids from skimmed buttermilk.

The objective of this study is to characterize the efficiency of the skimming of buttermilk and butter serum resulting from industry and to determine the consequences of the skimming on the lipid composition and structure (size distribution, microstructure) of related products. Simultaneously, this work improves knowledge on the microstructure of lipids in both buttermilks and butter serums using microscopy analyses.

2. Materials and methods

2.1. Industrial buttermilks and butter serums

Fresh industrial buttermilks and butter serums were provided by four French dairy industrial sites (kept confidential). Sweet buttermilks (n = 33) were produced at the industrial scale in a continuous way according to the NIZO process (Netherlands Dairy Research Institute, 1976). Industrial butter serums (n = 24) were obtained after melting (around 60–65 °C) and centrifugation of butters produced from the NIZO process. The composition of the buttermilks and butter serums were in the range of those reported in the literature for both industrial and laboratory scale products, either fresh or reconstituted from powder (Fauquant et al., 2014; Rombaut & Dewettinck, 2006; Vanderghem et al., 2010) (see Table 1 in Section 3, Results and discussion).

2.2. Skimming

Skimming of the industrial whole buttermilks and butter serums was carried out by centrifugation. At industrial scale, skimming was performed under traditional skimming conditions in a cream separator comprising conical disks. At pilot scale, skimming was carried out at a temperature of 60 °C (sometimes at 50 °C with no significant change in lipid composition of products after the skimming step) with a product flow of 500 L h^{-1} using a cream separator (Elecrem type 500, Fresnes, France). Samples before and after skimming were collected.

2.3. Determination of the skimming efficiency

In order to evaluate the proportion of lipids removed during the skimming step, the efficiency of skimming (%) is calculated as follows:

$$\text{Skimming Efficiency}\ (\%) = \left(\text{Lipid}_{\text{whole product}} - \text{Lipid}_{\text{skimmed product}}\right)/\text{Lipid}_{\text{whole product}} \times 100$$

where $\text{Lipid}_{\text{skimmed product}}$ and $\text{Lipid}_{\text{whole product}}$ are the concentrations of total lipid in g per kg of skimmed and whole liquid product respectively.

2.4. Chemical analyses

Except for lipid analyses, whole and skimmed buttermilks and butter serums were characterized in their liquid form. For lipid analyses, all samples were freeze-dried at −20 °C during 72 h with a lyophilizer CIRP CS 10-0.8 (Serail, La Coudray Saint Germer, France) and powders obtained were stored at −20 °C under vacuum.

Dry matter (DM) analyses were performed by drying about 5 g of sample at 103 °C for 7 h in a capsule containing sand (IDF, 1987). Protein content was determined using the Kjeldhal nitrogen determination method (IDF, 1993) with 6.38 as a nitrogen to protein conversion factor.

Total lipid content of lyophilized buttermilks and butter serums was determined by an adapted protocol of the cold extraction procedure developed by Folch, Lees, and Stanley (1957). This total lipid content

Table 1
Average composition of buttermilks and butter serums before and after the skimming step. Values are expressed in $g \cdot kg^{-1}$ of product (unless stated otherwise) ± standard deviation.

	Whole buttermilk	Buttermilk after the skimming step	Whole butter serum	Butter serum after the skimming step
Number of samples	33	19*	24**	27
Number of samples skimmed at pilot scale		13		24
Number of samples skimmed at industrial scale		6		3
DM ($g \cdot kg^{-1}$)	$87^{\underline{a},a} \pm 8$	$82^{\underline{a}} \pm 8$	$110^{\mathbf{a},b} \pm 8$	$102^{\mathbf{a}} \pm 10$
Proteins ($g \cdot kg^{-1}$)	$29^{\underline{a},a} \pm 3$	$27^{\underline{a}} \pm 3$	$36^{\mathbf{a},a} \pm 3$	$35^{\mathbf{a}} \pm 4$
Proteins (% of DM)	$33^{\underline{a},a} \pm 2$	$33^{\underline{a}} \pm 1$	$33^{\mathbf{a},a} \pm 3$	$35^{\mathbf{a}} \pm 1$
Total lipids ($g \cdot kg^{-1}$)	$9^{\underline{a},a} \pm 4$	$6^{\underline{b}} \pm 2$	$25^{\mathbf{a},b} \pm 8$	$19^{\mathbf{b}} \pm 3$
Total lipids (% of DM)	$10^{\underline{a},a} \pm 5$	$7^{\underline{a}} \pm 4$	$23^{\mathbf{a},b} \pm 5$	$19^{\mathbf{b}} \pm 2$
Polar lipids ($g \cdot kg^{-1}$)	$1.1^{\underline{a},a} \pm 0.1$	$1.0^{\underline{a}} \pm 0.2$	$9.7^{\mathbf{a},b} \pm 1.7$	$9.3^{\mathbf{a}} \pm 1.5$
Polar lipids (% of DM)	$1.2^{\underline{a},a} \pm 0.1$	$1.2^{\underline{a}} \pm 0.2$	$8.8^{\mathbf{a},b} \pm 1.1$	$8.9^{\mathbf{a}} \pm 1.1$
Polar lipids (% of lipids)	$14^{\underline{a},a} \pm 5$	$20^{\underline{b}} \pm 5$	$40^{\mathbf{a},b} \pm 7$	$49^{\mathbf{b}} \pm 6$

DM: dry matter;
Values are means of all the samples for a product type, regardless of the factory origin, the process and the way of skimming (laboratory scale or industrial).
Means in a row with different letters (a, b) are significantly different at $p < 0.001$. Statistical results are presented with letters in: italic for whole products (comparison between whole buttermilks and whole butter serums), underscore for buttermilks (comparison between whole and skimmed buttermilks), and bold for butter serums (comparison between whole and skimmed butter serums).
* Only 19 out of 33 whole buttermilks were skimmed and analyzed.
** The 3 whole butter serums corresponding to the skimmed butter serums provided by the industrial partners, were not analyzed.