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# Improved heat stability of recombined evaporated milk emulsions upon addition of phospholipid enriched dairy by-products

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ABSTRACT

Over the last decades, milk fat globule membrane (MFGM) fragments have gained a considerable attention for their beneficial technological and nutritional properties due to the presence of proteins and phospholipids. During butter production, the MFGM is ruptured and a great amount of membrane material migrates to the aqueous fraction, known as buttermilk. Its high phospholipid concentration attributes a very interesting functionality to buttermilk.

As it has been shown before that phospholipid addition may improve the heat stability of concentrated dairy emulsions during sterilization, the effect of two phospholipid enriched dairy by-products on the heat stability of recombined evaporated milk emulsions was investigated. To that end, a cream residue powder (CRP) originating from the production of butter oil from dairy cream, as well as a sweet buttermilk powder (SBP) have been used to reduce the undesirable changes taking place during intense heating of concentrated milk. Samples were prepared containing CRP or SBP in different concentrations (0–6 %) and were heated for multiple time intervals at sterilization conditions (121 °C). Both phospholipid enriched dairy by-products could largely reduce the pronounced viscosity increase as well as the increase in particle size observed upon intensive thermal treatment. Whereas the stabilizing effect of both products was directly proportional to their concentration, still the effect of CRP was more pronounced as compared to SBP: the addition of the maximum concentration (6%) of both products resulted in a similar particle size distribution and viscosity as compared to the original emulsion before heating, while a lower concentration of CRP (4%) also had a significant heat stabilizing effect. Whereas the difference in effectiveness could be probably related to the phospholipid content of both dairy ingredients, still it has to be kept in mind that these two ingredients not only differed in this aspect. Determination of the protein load revealed that phospholipid enriched dairy by-products reduced the increase in surface protein load upon sterilization, which points toward a reduced heat-induced interaction between the dairy proteins.

Overall, our experiments revealed that phospholipid enriched dairy by-products have interesting functional properties and largely improve the heat stability of recombined evaporated milk emulsions. For the two products considered, their effect seemed to be related to their phospholipid content.



## 1. Introduction

Buttermilk is the liquid product obtained during churning of cream in the butter making process. It contains all the water-soluble components of cream such as the milk proteins, lactose and minerals (Sodini, Morin, Olabi, & Jiménez-Flores, 2006; Vanderghem et al., 2010). Buttermilk has been used as animal feed or has been dried to be incorporated in bakery products due to its positive impact on flavor (Vanderghem et al., 2010). However, over the years it gained a considerable potentiality because of its high content in milk fat globule membrane (MFGM) material, a product rich in phospholipids (Corredig & Dalgleish, 1997; Rombaut, Dejonckheere, & Dewettinck, 2007). The MFGM is disrupted during churning and migrates into the buttermilk portion. In a quite similar way, an MFGM-enriched side stream is also obtained during butter oil production by mechanical treatment to induce phase separation, and centrifugation of concentrated dairy cream.

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In this case too, the MFGM becomes displaced to the aqueous phase due to the mechanical stress. According to Patton and Keenan (1975), the phospholipid fraction of the MFGM consists of phosphatidylethanolamine (PE), phospatidylcholine (PC), phospatidylserine (PS) and phosphatidylinositol (PI). Due to their enhanced biological activity and their positive effects on health, phospholipids have been extensively used for several medicinal studies (Britten, Lamothe, & Robitaille, 2008), e.g. related to cell growth and development, prevention of Alzheimer's disease (Spitsberg, 2005), or protection against bacterial toxins and infections (Sprong, Hulstein, & van der Meer, 2002). It is also known that phospholipids possess remarkable emulsifying properties, making buttermilk a very interesting functional dairy product (Corredig & Dalgleish, 1998; Elling, Duncan, Keenan, Eigel, & Boling, 1996; Wong & Kitts, 2003).

Heat treatment is a very important unit operation during milk processing. However, upon heating at elevated temperatures, heat coagulation takes place as a result of whey protein denaturation (Jeurnink & Dekruif, 1993; Singh, 2004), whereby heat induced whey protein interactions seem to play a crucial role. More precisely, at temperatures higher than 70 °C, β-lg undergoes several conformational changes, making the hydrophobic residues as well as the free sulfhydryl group accessible (Galani & Apenten, 1999; Hansted, Wejse, Bertelsen, & Otzen, 2011; Tran Le et al., 2011). The addition of phospholipids has been proved to protect milk against heat coagulation upon severe heating (Chen & Dickinson, 1998; Chen, Dickinson, Langton, & Hermansson, 2000; McCrae & Muir, 1992; Tran Le et al., 2007). Hereby, it either displaces the protein from or it interacts with the protein at the interface (Brown, Carroll, Pfeffer, & Sampugna, 1983; Kristensen, Nylander, Paulsson, & Carlsson, 1997; McCrae, 1999).

In this contribution, the effect of two MFGM enriched and hence phospholipid enriched dairy by-products on the heat stability of recombined evaporated milk was evaluated. Heat induced effects were derived from both viscosity and particle size analyses, whereas protein surface load determinations were performed to obtain a better understanding of the effect of the MFGM-enriched dairy products on protein interactions in the recombined evaporated milk.

## 2. Materials and methods

### 2.1. Materials

High-heat skimmed milk powder (SMP), sweet buttermilk powder (SBP), and cream residue powder (CRP) were obtained from FrieslandCampina (Deventer, The Netherlands). According to the manufacturer, the SMP contained 37.3% (w/w) protein, 0.5% (w/w) fat and 0.15% (w/w) phospholipids. SBP contained 32.0% (w/w) protein, 9.0% (w/w) fat and 2.9% (w/w) phospholipids, whereas the CRP sample contained 30.2% (w/w) protein, 15.0% (w/w) fat and 6.4% (w/w) phospholipids.

The high oleic sunflower oil (Hozol, Contined, The Netherlands) contained maximum 0.05% free fatty acid as oleic. Its melting point is at 0 °C and hence the oil remains clear even after 10 h storage at 4 °C.

### 2.2. Emulsion preparation

Recombined evaporated milk emulsion samples were prepared containing 16.5% (w/w) SMP and 6.5% (w/w) oil. SBP or CRP were added in concentrations ranging from 0 to 6 %. Hereby, the composition of the samples was adjusted to ensure that they all contained the same amount of protein and fat (Table 1). Milk powders were diluted into 0.02% $NaN_3$ (Acros Organics) aqueous solution aiming to prevent microbial contamination, whereas Hozol oil was added as the dispersed phase. The samples were pre-homogenized by an IKA Ultra-Turrax TV45 (Janke & Kunkel, Staufen, Germany) for about 1 min and homogenized by a Microfluidiser 110S (Microfluidics Corporation, Newton, Massachusetts, USA) having its heat exchange coil immersed in a water bath at 55 °C. The samples were microfluidized at a compressed air pressure of 2 bar, corresponding to a liquid pressure of about 280 bar for 2 min. The pH of the samples was determined (HI 4222, Hanna instruments) after homogenization, and was approximately 6.52 for all samples.

**Table 1**
Calculated composition of recombined milk model systems with partial replacement of SMP by 2.0%, 4.0% and 6.0% (w/w) sweet butter milk powder and cream residue powder, respectively, in order to have the same oil and protein content as in an emulsion containing 6.5% (w/w) Hozol oil and 16.5% (w/w) high heat skimmed milk powder. The last line (in italics) contains the calculated phospholipid content, as derived from the PL content of the SMP, SBP and CRP powders used.

| Composition (% w/w) | | | | | | | |
|---|---|---|---|---|---|---|---|
| Components | Reference | +Sweet buttermilk powder (%) | | | +Cream residue powder (%) | | |
| | 0.0 | 2.0 | 4.0 | 6.0 | 2.0 | 4.0 | 6.0 |
| Skim milk powder | 16.5 | 14.7 | 13.0 | 11.2 | 15.1 | 13.7 | 12.3 |
| Hozol oil | 6.5 | 6.3 | 6.1 | 5.9 | 6.1 | 5.8 | 5.4 |
| $NaN_3$ aqueous solution | 77.0 | 77.0 | 76.9 | 76.8 | 76.8 | 76.6 | 76.3 |
| *Phospholipid content* | *0.02* | *0.08* | *0.14* | *0.19* | *0.15* | *0.28* | *0.40* |

### 2.3. Heat treatment

Prior to heat treatment, a volume of 10 ml of each sample was transferred into 20 ml headspace vials (75.5 × 22.5 mm, 1st hydrolytic class, DIN-crimp neck, long neck, HC-bottom; caps: 20 mm combination seal: aluminum cap, plain, with center hole, silicone transparent blue/PTFE white, 35° shore, 3.0 mm) which were clipped on a platform and positioned in a temperature-controlled oil bath (Fritel turbo SF®, 5 L capacity) in order to be heated at 121 °C for a time range of 0–15 min.

In order to optimize the heating process, preliminary measurements were performed including samples in the absence of phospholipid-enriched dairy products. During heating, the temperature inside the sample was measured by using thermocouples (type K) connected with an electronic digital thermometer (Agilent 34970A). According to the recorded temperature profiles, it took about 9 min before the whole contents reached a temperature of 121 °C. Hence, in order to evaluate the influence of sterilization temperature on recombined evaporated milk samples with and without phospholipids, the heating time was considered from the time that the temperature was homogeneous inside the sample. Therefore, the maximum heating time of 15 min at 121 °C actually involved submersion of the glass vials for 24 min, i.e. 9 min equilibration to reach 121 °C and 15 min holding at 121 °C.

### 2.4. Viscosity measurements

A Programmable LV-DV-II + Viscometer (Brookfield, USA), at speeds ranging from 0 to 200 RPM (rotations per minute) was used to measure the viscosity of samples before and after heating. All measurements were performed at room temperature. Spindle 21 (with an 8 ml small volume adapter) has been used to measure the viscosity of samples which retained their liquid structure, whereas spindle 34 (immersed directly into the glass vials) has been used to measure the viscosity of samples which obtained a gel-like structure. The conversion of RPM into shear rates ($s^{-1}$) is performed by multiplying the value of the rotational speed with the shear rate