

Fractionation of globular milk fat by membrane microfiltration

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Abstract — Separation of milk fat in small globules (SG) (diameter lower than 2 μm) and in large globules (LG) (diameter higher than 2 μm) was realized by a patented process using special ceramic microfiltration membranes. Transformations in drinking milks, yoghourts, sour cream, camembert, Swiss cheese and butters were realized from milks of which the fat content was adjusted either by reference cream or by creams issued from the SG or the LG fractions. Significant differences in texture and organoleptic properties were found in the manufactured products. Except for butter, use of milks containing small fat globules led to more onctuous products and more finer textural characteristics versus products made with reference creams or with mainly large fat globules. Fat globule membrane appeared to have a high ability to bind water in cheese and so, to allow a significant increase in cheese yield.

microfiltration / fat globule size / texture / dairy product

1. INTRODUCTION

In milk, fat is predominantly present in spherical globules varying in diameter from 0.1 μm to 15 μm [10]. Their number per mL is between 10^{10} to 10^{11} and they develop a surface area in the range of 5 to 11 m^2 per 100 g of milk [9]. Globules below 1 μm in diameter account for 80% or more of the total globule number, but they contain little of the total volume of milk fat. Globules between 1 and 8 μm in diameter contain 90% or more of the total volume of milk fat [2].

Milk fat globules are individually surrounded by a membrane which maintains their integrity and renders them compatible with the aqueous environment [7]. Milk fat globules membranes (MFGM) composition appears very complex and its structure is still poorly understood [10]. Available information on gross composition of MFGM shows well this uncertainty. MFGM comprises from about 2 to more than 6% of the mass of globules. Proteins, at least 10 major species would represent 25 to 60% of the MFGM material. Most of them, including the main specific butyrophilin, would be

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glycosylated [6]. At least 25 different enzymatic activities have been found to be associated with MFGM [6]. According to Walstra [10], the MFGM average thickness would be 15 nm and would represent $0.4 \text{ g}\cdot\text{L}^{-1}$ of milk. On the other hand, the complex material of MFGM leads to a zeta potential of -12 mV and an isoelectric pH of the washed milk fat globules of 3.7.

It is well known that a strong increase of the fat globule number by homogenization modifies the mouth feeling of a liquid milk. For the same fat content, homogenized milk appears sweeter and has more body than the reference milk [1]. Moreover, it can be supposed that a large part of the microstructure and consistency of dairy products such as cheeses is largely affected by interactions between the casein matrix and fat globule membranes. Consequently, a possible way to increase or to decrease these interactions could be to modify, in the milk to be processed, the number of fat globules, for a given fat content.

Moreover, it could also be considered that an increase of the fat globule number i.e. to prepare a milk with small fat globules, would have other effects due to the ability of the MFGM to bind water [4] and the difference in fatty acid composition between large and small fat globules [8]. Such an adjustment of fat globule number could not be done by homogenization because this mechanical treatment disrupts the MFGM integrity and often leads to detrimental consequences such as accelerated lipolysis. Among the separation technologies known for not damaging MFGM and which could allow separation of fat globules according to their size, centrifugation [8] and membrane microfiltration (MF) are conceivable; the latter was preferred since we thought that fat globule size could be a much better separation criterion between small and large globules than fat globule density. Consequently, a patented process [5] was developed to fractionate milk fat

globules from whole milk and creams by MF with special ceramic membranes.

This paper reports the preliminary results observed on dairy products made from milks of which the fat portion was composed either by large globules (LG) (diameter higher than $2 \mu\text{m}$) or by small globules (SG) (diameter lower than $2 \mu\text{m}$).

2. MATERIALS AND METHODS

2.1. Dairy products

2.1.1. Milk

Raw whole milk was obtained from a dairy plant (CLE, 35360 Montauban-de-Bretagne, France).

2.1.2. Cream preparation

– Reference cream was obtained by separation of raw whole milk heated to $47 \text{ }^\circ\text{C}$ in an Elecrem[®] separator (Elecrem “3” – Sté Elecrem, 92174 Vanves, France).

– Small globule creams were obtained by separation of the MF permeates with the same Elecrem[®] equipment at $47 \text{ }^\circ\text{C}$. Fat content ($350 \text{ g}\cdot\text{kg}^{-1}$) was adjusted with skim-milk.

– All dairy products were cooled and stored at $2 \text{ }^\circ\text{C}$ before use.

2.2. Membrane microfiltration

All membrane microfiltration separations of large and small globules were realized at $50 \text{ }^\circ\text{C}$ as described in INRA patent [5].

2.3. Heat treatments

Heat treatments of milks to be transformed into consumption milk, yoghourts, cheeses, sour creams and butter were realized in an Actijoule[®] heating equipment (Pilot Plant 1959, Sté Actini-France, 74500 Evian, France).

2.4. Fat standardization of processed milks

Except for the SG drinking milk, processed milks were prepared as follows: reference milks by mixtures of skim milk and reference cream; SG milks by mixtures of SG creams with skim milk; LG milks by mixtures of LG creams with skim milk.

2.5. Analysis

Total solids were obtained by weighting before and after drying in an oven during 7 h at 102 (± 2) °C.

Fat content was determined by using acido-butyrometric methods of Gerber and Van Gulik apart from milks for which DairyLab® (Foss Electric, Nanterre, France) measurements were used.

2.6. Rheology

Rheological characteristics of yoghurts, fresh cheeses, sour creams, camembert and mini-Swiss cheeses were determined using a Stevens (LFRA, Lhomargy, 94208 Ivry-sur-Seine, France).

Those of butters by using an Instron Universal testing machine Model 4501 (Instron Ltd., High Wycombe, England) with a 10 mm penetration depth.

2.7. Tasting panel

The tasting panel consisted of 6 to 10 trained members depending on the tests.

3. RESULTS AND DISCUSSION

3.1. Membrane microfiltration performances

Over the large range of membrane pore size tested only results obtained specifically with a ceramic membrane of 2 μm average pore size diameter are reported.

With whole milk (fat content 39 $\text{g}\cdot\text{kg}^{-1}$), a volumic reduction ratio (VRR) of 8 was obtained at 50 °C. In these conditions, the permeate flux was 700 $\text{L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and fat contents of the permeate and of the retentate were respectively 17 $\text{g}\cdot\text{kg}^{-1}$ and 197 $\text{g}\cdot\text{kg}^{-1}$. Increasing the fat content of the inlet product to 120 $\text{g}\cdot\text{kg}^{-1}$ decreased the VRR to 4.1 and the permeate flux to 250 $\text{L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ but increased the fat content of the permeate and of the retentate to 69 $\text{g}\cdot\text{kg}^{-1}$ and 297 $\text{g}\cdot\text{kg}^{-1}$ respectively.

3.2. Drinking milk assays

Semi skim milks were prepared by a mixture of raw skim milks and reference creams and compared with MF permeates directly issued from the treatment of raw whole milks. Both products with the same fat content (17 $\text{g}\cdot\text{kg}^{-1}$) were then heated to 72 °C –15 s and cooled down to 2 °C before to be tasted.

Unanimously, the tasting panel found the MF permeates (SG milks) significantly more onctuous and more creamy than the reference milks.

3.3. Yoghurt assays

Six batches of milks were prepared: two references of which the fat content was adjusted either to 41 $\text{g}\cdot\text{kg}^{-1}$ or to 10.5 $\text{g}\cdot\text{kg}^{-1}$ by adding the appropriate amount of reference cream to skim milk; two SG milks by addition of SG cream, two LG milks by addition of LG cream to the same skim milk. All batches were then heated to 94 °C – 4 s, cooled down to 42 °C and added with 0.08 $\text{unit}\cdot\text{kg}^{-1}$ of Ezal MY 800 starters (Rhodia-Textel, 86220 Dangé, St-Romain, France) for 6 h. After cooling to 4 °C, penetrometry tests were performed. Full fat yoghurt shear stress was found to be 507 Pa for the reference, 607 Pa for the LG and 404 Pa for the SG. The same rheological measurements

for the low fat yoghourts were respectively: 669 Pa for the reference product, 810 Pa for the LG and 601 Pa for the SG.

These rheological differences were also detected by the tasting panel who qualified the SG yoghourts smoother and finer in texture than the reference products. On the contrary, the in-mouth texture of LG products was found firmer and grainy compared to that of the references.

3.4. Fresh cheese assays

Three batches of milks were prepared by mixing the requested amounts of skimmilk either with reference cream or with the MF 0.2 μm retentate (LG products) or with the MF 0.2 μm permeate (SG products) in order to obtain the same fat content of 41 $\text{g}\cdot\text{kg}^{-1}$. All milks were then heated to 94 °C – 4 s, cooled down to 22 °C, added with 0.08 $\text{unit}\cdot\text{kg}^{-1}$ of Ezal-MM 101 starters (Rhodia-Textel) and 0.01 mL of rennet extract (SKW Biosystems, 21201 Beaune, France) per kg. Eighteen hours after renneting, drainage on clothes was done and each curd was homogenized in an ALM equipment (model Lab – Ets Auguste et Des Moutis) using a D 190 screw.

The three lots of fresh unripened cheeses with the same total solids (186 $\text{g}\cdot\text{kg}^{-1}$) and fat (78 $\text{g}\cdot\text{kg}^{-1}$) contents were then rheologically characterized and tasted. The same shear stress (450 Pa) was determined on reference and on LG product but SG product had a lower shear stress (387 Pa). No taste differences were detected by the tasting panel between the three products. However in mouth texture of SG fresh cheese was significantly appreciated as smoother and finer than reference and LG products.

3.5. Sour cream assays

Reference, LG and SG creams prepared as described above (Material and Methods) were adjusted to 350 $\text{g}\cdot\text{kg}^{-1}$ fat, heated to 72 °C – 15 s and cooled down to 22 °C.

Then, they were added with 0.1 $\text{unit}\cdot\text{kg}^{-1}$ of Ezal-MM 100. After 17 h, all products were cooled down at 4 °C and rheologically characterized. Shear stress was 3040 Pa for cream, 3590 Pa for LG cream and 258 Pa for SG cream.

3.6. Camembert cheese assays

Cheese milks were prepared by mixing either reference cream or SG cream with the appropriate amount of the same skim-milk in order to reach a fat content of 28 $\text{g}\cdot\text{kg}^{-1}$. Both fat standardized cheese milks were heated to 72 °C – 15 s, cooled down to 34 °C and added with 0.02 $\text{unit}\cdot\text{kg}^{-1}$ of MM 101 Ezal starters and 0.08 $\text{unit}\cdot\text{kg}^{-1}$ of Flora Danica (Hansen-France, 91292 Arpajon, France). When pH reached 6.25, rennet extract 0.2 $\text{mL}\cdot\text{kg}^{-1}$ (SKW Biosystems, 21201 Beaune, France) was added and both curds were cut, drained and cooled down according to the same usual cheese-making procedures.

SG cheeses TS content were significantly lower (39.3 g p. 100) than that of reference cheeses (40.1 g p. 100). This TS content difference corresponded to a 2% increase in cheese yield.

This TS difference was reflected by the rheological characteristics. Firmness and shear stress of reference camembert were respectively 36.0 N and 5838 Pa versus 31.6 N and 5130 Pa for the SG products.

Firmness difference was still detected at 25 days of ripening by the tasting panel that qualified SG cheeses core as less chalky than the reference one.

3.7. Mini Swiss cheese assays

Cheese milks were prepared with reference, LG and SG creams mixed with skim-milk, thermized at 65 °C – 20 s and manufactured in mini-Swiss cheeses as described by Buisson et al. [3]. No significant differences were noticed in the cheese making processes nor in acidification kinetics.

At 20 h, the three cheeses showed the same fat / TS ratio (0.45) but the TS content was respectively 61.2 g p. 100 for the reference product, 58.6 g p. 100 for the SG product and 60.6 g p. 100 for the LG mini-cheese. Shear stress characterization confirmed these differences; the determined values were respectively 58 500 Pa for the reference product, 44 500 Pa for the SG product and 57 600 Pa for the LG mini-cheese. A 3% increase in cheese yield was observed for the SG cheese versus the reference or the LG cheeses. At 60 days of ripening in the conditions described by Buisson et al. [3] the differences in TS of the three cheeses still remained: 63 g p. 100 for the reference product, 62.4 g p. 100 for the LG product and 60.9 g p. 100 for the SG mini-cheese.

No difference was found by the tasting panel between the reference and the LG cheeses at this ripening age, but the SG cheese was judged smoother and more onctuous than the two other cheeses.

3.8. Buttermaking assays

Reference, LG and SG creams were prepared as described above (Material and Methods). They were then heated to 75 °C –20 s and cooled down to 5 °C. The following day, they were gently heated to 18 °C and added with Ezal starters (0.05 unit per kg of Ezal MM 100 and 0.05 unit per kg of Ezal MD 099). Seventeen hours after, when pH reached 5.2–5.3, all creams were cooled to 12 °C and churned as usually practiced. No difference in churning abilities was noticed.

The composition of reference and LG butters was similar: fat 85.1 and 85.0 g p. 100 respectively, water 12.3 and 12.1 g p. 100, salt 1.03 and 1.08 g p. 100. The tasting panel judged with a slightly higher score the LG butter versus the reference one. LG butter was found to have a more onctuous and more melting in-mouth texture. Such a rheological behavior was confirmed by the penetrometry characterization: compression

strengths were respectively 125 N for the reference and 104 N for the LG butter.

On the contrary, SG butter had a higher fat content (88.1 g p. 100), lower water and salt contents (10.4 g p. 100 and 8.9 g p. 1000) and it was characterized by the tasting panel as greasy and oily.

4. CONCLUSION

These results suggest that increasing or decreasing the number of fat globules while keeping native their membrane leads to significant changes in dairy products texture. Such changes are likely induced by the intensity of the interactions between the milk fat globule components and their environment i.e. water components, soluble components, casein micelles and casein matrix. The described results are preliminary and further work is requested in many fields. Fat globules separation by membrane microfiltration has to be optimized for increasing performances or for producing different size subpopulations of milk fat globules than those studied in this work. Moreover, shear stress caused by the pumps, the valves, the pipes carried out in the used MF rig has to be minimized. Potential differences in triglyceride composition of SG and LG fractions have to be determined. Many other research areas can be considered e.g., in cheese biochemistry or concerning functional properties of dairy products.

Nevertheless, we can conclude that thanks to the use of membrane microfiltration, a new possibility to adjust texture and maybe flavor of dairy products is open. Acting on the fat globule size, without damage of the MFGM offers a way to understand deeper how fat globules play a role in textural characteristics of the different cheese varieties.

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REFERENCES

- [1] Alais C., Science du lait, Principes des techniques laitières, 4^e éd., Sepaic, Paris, 1984.
- [2] Brunner J.R., Physical equilibria in milk: the lipid phase, in: Webb B.H., Johnson A.H., Alford J.A. (Eds.), Fundamentals of Dairy Chemistry, Avi Publishing Company Inc., Westport, 1974, pp. 474–602.
- [3] Buisson U., Kerjean J.R., Courroye M., Miniaturisation de la fabrication fromagère : un nouveau test pour mesurer l'aptitude fromagère des laits, Tech. Lait. Mark. 1024 (1987) 17–23.
- [4] Fennema O., Powrie W.D., Fundamentals of low-temperature food preservation, in: Chichester C.O., Mrak E.M., Stewart G.F. (Eds.), Advances in Food Research, Vol. 13, Academic Press, New York, 1952, pp. 219–347.
- [5] Goudédranche H., Maubois J.L., Fauquant J., Fr. Patent 2 776 208, 1998.
- [6] Keenan T.W., Dylewski D.P., Intra cellular origin of milk lipid globules and the nature and structure of the milk globule membrane, in: Fox P.F. (Ed.), Advanced Dairy Chemistry, Vol. 2, Lipids, 2nd ed., Chapman & Hall, London, 1995, pp. 89–130.
- [7] Mulder H., Walstra P., The milk fat globule: emulsion science as applied to milk products and comparable foods, Centre for Agricultural publishing and documentation, Wageningen, The Netherlands, 1974.
- [8] Timmen H., Patton S., Milk fat globules: fatty acid composition, size and in vivo regulation of fat liquidity, Lipids 7 (1988) 685–689.
- [9] Walstra P., Physical chemistry of milk fat globules, in: Fox P.F. (Ed.), Advanced Dairy Chemistry, Vol. 2, Lipids, 2nd ed., Chapman & Hall, London, 1995, pp. 131–178.
- [10] Walstra P., Geurts T.J., Noomen A., Jellema A., van Boekel M.A.J.S., Dairy Technology: principle of milk properties and processes, Marcel Dekker, New York, 1999.