Journal of Food Technology 12 (2): 54-57, 2014
ISSN: 1684-8462

Impact of Microfiltration for Bacteria Removal on Milk Constituents

Ninane Veronique, Grelet Clement, Romnee Jean-Michel and Dehareng Frederic
Walloon Agricultural Research Center, 24 Chaussee de Namur,
B-5030 Gembloux, Belgium

Abstract: The purpose of this study was to measure the impact of microfiltration for bacteria removal on the principal chemical components of milk. Six skimmed raw milks were micro-filtered using a laboratory bench unit fitted with a Membralox® ceramic membrane having a pore size of 1.4 μm. Microfiltration resulted in a significant reduction in crude protein ($p = 0.001$), casein ($p = 0.008$), calcium ($p = 0.002$), phosphorus ($p = 0.045$) and magnesium ($p = 0.016$) contents of milk. While, crude proteins and casein accumulated in the retentate, the minerals seemed to be entrapped by the membrane. In contrast to these constituents, potassium, sodium and lactose contents remained statistically unchanged in milk.

Key words: Microfiltration, milk, proteins, minerals, sodium

INTRODUCTION

Microfiltration is a mechanical method of separating particles from a liquid that is used in particular in the sanitary processing of milk. The milk is filtered through a porous ceramic membrane which retains bacteria, spores and somatic cells and lets other constituents through with the exception of fat which is eliminated beforehand and treated separately. As a non-thermal process, it has the advantage of treated raw milk for sanitation without denaturing heat-sensitive molecules, thereby, preserving the original organoleptic and nutritional qualities of the milk. It has the additional advantage for cheese-making of eliminating butyric spores from the milk: these may impair some cheeses, particularly hard pressed cheeses.

The pore size of the membrane determines the size of the particles that will be retained; for the purpose of bacteria removal, membranes used generally have a pore size of 1.4 μm (Jost and Jelen, 1997; Saboya and Maubois, 2000; Te Giffel and van der Horst, 2004). The effectiveness of these membranes has been widely proven: they retain between 99.84 and 99.99% of milk bacteria (Pafylias *et al*., 1996; Elwell and Barbano, 2006) including contaminant species and their sporulated forms which are eliminated to a level of between 99.93 and 99.99% (Olesen and Jensen, 1989; Trouve *et al*., 1991; Madec *et al*., 1992). At the same time, somatic cells which have a larger cell volume than bacteria, are retained in their entirety (Hoffmann *et al*., 2006).

The effect of microfiltration for bacteria removal on the other constituents of milk, particularly proteins and minerals, is less documented. Saboya and Maubois (2000) reported a rate of protein permeation in excess of 99.0%, while Pafylias *et al*. (1996) concluded that there was no significant loss and Hoffmann *et al*. (2006) drew the conclusion that the loss was minimal, however, the losses are rarely precisely quantified.

MATERIALS AND METHODS

Microfiltration equipment: The equipment used was a laboratory bench set-up comprising a Quattroflow 1200 S pump (Membracon Process Separation, Oss, Netherlands), a 10 L thermo-regulated stainless steel tank (Karma Global, Taiwan) and a stainless steel housing (Tami Industries, Nyons, France) used with membranes of 10 mm in diameter and 25 mm in length. For ease of organization of sampling and permeate flow control, one of the two permeate outlets from the housing was sealed. The circuit was also fitted with three manometers (accurate to 0.2 bars) and a temperature probe (Fig. 1).

The unit was fitted with a Membralox® ceramic membrane with an average pore size of 1.4 μm (Pall Exekia, Bazet, France).

Experimental conditions: Microfiltration tests were performed with raw milk skimmed to 0.07% fat and kept at a temperature of 50°C. Initial volumes of about 7 L of skimmed milk were circulating with a tangential flow of 5.7 m sec^{-1} while the trans-membrane pressure was maintained at about 0.6 bars.

Corresponding Author: Ninane Veronique, Walloon Agricultural Research Center, 24 Chaussee de Namur, B-5030 Gembloux, Belgium

Six bulk raw milks collected at different times were micro-filtered by this way. In each case, four successive 250 mL samples of micro-filtered milk were collected at the permeate outlet and at the same time, four successive samples of raw milk were taken in the thermo-regulated storage tank.

The permeate flow rate was measured on the occasion of sampling the micro-filtered milk. The permeate flow rate reduced between the first and the fourth (and last) sample from, in average, 7.3-6.3 L h^{-1}; revealing a beginning of membrane clogging.

The absence of flora and cells quantifiable from the micro-filtered milk, using the Bactoscan FC and Fossomatic (Foss, Hillerod, Denmark) Methods, respectively was used to check the effectiveness of sanitation.

Cleaning: The unit was rinsed between tests with demineralised water then cleaned by circulating a warm solution (over 50°C) of NaOH (20 g L^{-1}) and bleach (3%) through it for 45 min. After rinsing with demineralised water, the final stage of cleaning was to circulate a warm solution (over 50°C) of HNO_3 (58%) through it for 15 min. The treatment was completed by rinsing the unit with demineralised water that was previously sterilized at 120°C for 20 min.

The unit was rinsed again before use with demineralised water and then emptied as completely as possible, using a pump. The residual volume of water left in the circuit was of 110 mL.

Analysis: Analyses were carried out on samples preserved at -20°C. For crude protein and casein contents, nitrogen was determined in the milk (N) and in the supernatant left after precipitation of the caseins at pH 4.6 (NCN) by the Kjeldahl Method using a Tecator unit (Humeau, Nantes, France). The crude protein content was equal to N×6.38 and the casein content was equal to the difference between the two sources of nitrogen (N-NCN)×6.38.

Calcium, phosphorus, potassium, magnesium and sodium contents were determined by inductively coupled plasma atomic emission spectroscopy (Ultima ICP-AES from Horiba Jobin-Yvon, Longjumeau, France) following a protocol adapted by Murcia *et al.* (1999). Lactose content was assessed using differential pH measurement (ISO 26462:2010 Geneva, Switzerland).

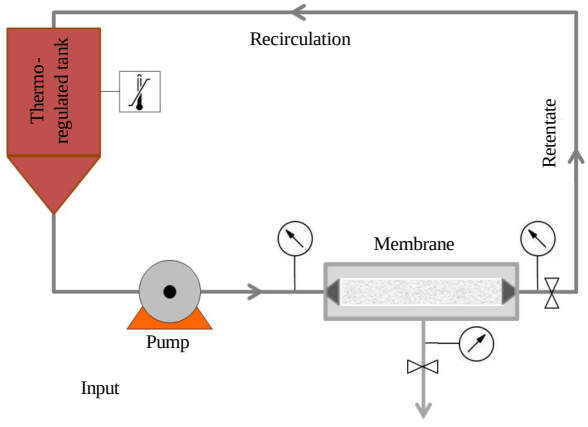

Fig. 1: The microfiltration unit

Statistical treatments: Equality of means was tested using a two-way analysis of variance with or without repetition of the experiment depending on whether the requirement was to prove the impact of microfiltration or of sampling on the analytical parameters measured (Excel, Microsoft, Redmond, USA).

RESULTS AND DISCUSSION

The average content of most of the constituents measured in the milk had decreased after passing through the membrane (Table 1). The membrane retained ($p<0.05$) both nitrogen fractions and the minerals with the exception of potassium and sodium for which contents were unchanged further to microfiltration. The average rates of retention for crude protein, casein, phosphorus and magnesium were 1% while the average rate of retention for calcium was 2%. As with potassium and sodium, lactose was fully recovered in the micro-filtered milk (Table 1).

Four successive samples of raw milk were taken from the feeding tank which also collected the microfiltration retentate (Fig. 1). The raw milk was observed to have been enriched in crude protein and casein between the first and the fourth sample ($p<0.05$) in

Table 1: Composition of raw and micro-filtered skimmed milks

Composition	Raw skimmed milk (g kg^{-1})	Micro-filtered skimmed milk (g kg^{-1})	p-values
Crude protein	32.7±0.6	32.4±0.6	0.001
Casein	25.3±0.6	25.1±0.5	0.008
Calcium	1.19±0.03	1.17±0.04	0.002
Phosphorus	1.10±0.03	1.09±0.04	0.045
Magnesium	0.102±0.002	0.101±0.003	0.016
Potassium	1.60±0.02	1.58±0.04	0.080
Sodium	0.38±0.01	0.38±0.02	0.844
Lactose	50.7±0.7	50.9±0.08	0.397

Mean values±standard deviation from 24 samples. Significant difference between raw skimmed milk and micro-filtered skimmed milk if $p<0.05$

both cases, the mean enrichment for the six micro-filtered milks was 1%. However, no enrichment was shown between these two samples for the other parameters measured.

A reduction in crude protein and casein contents of skimmed milk was observed further to microfiltration testing. These observations are consistent with the data found in the literature: the occurrence of protein retention in the process of milk sanitation by microfiltration is reflected in data from Vincens and Tabard (1988), Schuck *et al.* (1994), Pafylias *et al.* (1996) and Hoffmann *et al.* (2006). Moreover, the average rate of crude protein retention was equivalent to the rates that could be deduced from the limited data obtained by Schuck *et al.* (1994) and Hoffmann *et al.* (2006) using pilot units fitted with a membrane of the same type as that used in this study.

At the same time, microfiltration had significantly depleted the milk in some minerals: calcium, phosphorus and magnesium. The effect of microfiltration for sanitation on the mineral constituents of milk is not well-documented; limited data, concerning calcium content of respectively single milk and two milks are given by Schuck *et al.* (1994) and by Hoffmann *et al.* (2006). In both studies, microfiltration has little or no impact on this parameter.

Calcium, phosphorus and magnesium are minerals which are partially bound to casein and could have been retained with it in the retentate. However, in contrast to observations in respect of crude protein and casein, the mineral depletion of the filtrate did not result in a concomitant enrichment of the retentate, suggesting that these minerals may have been trapped on or in the membrane. In their study of clogging in microfiltration membranes, Vetier *et al.* (1986) showed that calcium and phosphorus and also casein micelles, may be adsorbed by the alumina grains that form the membrane.

In the process of clogging, the ratio of calcium (or phosphorus) to nitrogenous matter is higher in the deposits accumulated on the alumina grains than in the casein micelles, suggesting that a proportion of the minerals adsorbed account for the soluble phase of milk. A compensatory effect of the adsorption of soluble minerals in respect of the retention of casein-linked minerals could be the reason for the lack of retentate enrichment observed in this study. In this study, although, being not drastic, clogging started rendering possible that particles were trapped in or on the membrane.

Lastly, the lack of any impact of microfiltration on the lactose content of milk is consistent with the data from Pafylias *et al.* (1996).

CONCLUSION

The purpose of this research was to measure the rate at which the principal chemical components of milk are retained by a membrane commonly used in the sanitary processing of milk via microfiltration.

REFERENCES

Elwell, M.W. and D.M. Barbano, 2006. Use of microfiltration to improve fluid milk quality. J. Dairy Sci., 89: E10-E30.

Hoffmann, W., C. Kiesner, I. Clawin-Radecker, D. Martin and K. Einhoff *et al.*, 2006. Processing of extended shelf life milk using microfiltration. Int. J. Dairy Technol., 59: 229-235.

Jost, R. and P. Jelen, 1997. Cross-flow microfiltration-an extension of membrane processing of milk and whey. Bull. Int. Dairy Fed., 320: 9-15.

Madec, M.N., S. Mejean and J.L. Maubois, 1992. Retention of *Listeria* and *Salmonella* cells contaminating skim milk by tangential membrane microfiltration (Bactocatch process). Lait, 72: 327-332.

Murcia, M.A., A. Vera, M. Martinez-Tome, A. Munoza, M. Hernandez-Cordoba and R. Ortiz-Gonzalez, 1999. Fast determination of the Ca, Mg, K, Na and Zn contents in milk and nondairy imitation milk using ICP-AES without mineralization stage. LWT-Food Sci. Technol., 32: 175-179.

Olesen, N. and F. Jensen, 1989. Microfiltration. The influence of operation parameters on the process. Milchwiss, 44: 476-479.

Pafylias, I., M. Cheryan, M.A. Mehaia and N. Saglam, 1996. Microfiltration of milk with ceramic membranes. Food Res. Int., 29: 141-146.

Saboya, L.V. and J.L. Maubois, 2000. Current developments of microfiltration technology in the dairy industry. Lait, 80: 541-553.

Schuck, P., M. Piot, S. Mejean, J. Fauquant, G. Brule and J.L. Maubois, 1994. Deshydratation des laits enrichis en caseine micellaire par microfiltration; comparaison des proprietes des poudres obtenues avec celles d'une poudre de lait ultra-propre. Lait, 74: 47-63.

Te Giffel, M.C. and H.C. van der Horst, 2004. Comparison between bactofugation and microfiltration regarding efficiency of somatic cell and bacteria removal. Bull. Int. Dairy Fed., 389: 49-53.

Trouve, E., J.L. Maubois, M. Piot, M.N. Madec and J. Fauquant *et al*., 1991. Retention de differentes especes microbiennes lors de l'epuration du lait par microfiltration en flux tangential [Retention of different microbial species in the l'treatment of milk by microfiltration in tangential flow]. Lait, 71: 1-13.

Vetier, C., M. Bennasar, B.T. de la Fuente and G. Nabias, 1986. Etude des interactions entre constituants du lait et membranes minerales de microfiltration [Study of interactions between milk constituents and mineral microfiltration membranes]. Lait, 66: 269-287.

Vincens, D. and J. Tabard, 1988. L'elimination des germes bacteriens sur membranes de microfiltration [The elimination of bacterial germs on microfiltration membranes]. Tech. Lait, 1033: 62-64.