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Original article

Quarg-making by ultrafiltration using polymeric and mineral membrane modules: a comparative performance study

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Summary — The present study was conducted to evaluate the performance of Hollow Fiber (HF-43 and 60), Carbosep S7 and Ceraver (P-19-40) modules for ultrafiltration of coagulated skim milk at pH 4.6 for making quarg. Three basic parameters, *ie* permeate flux, energy consumption/m^3 permeate and retention coefficients of total solids, protein, lactose and calcium, were taken for comparative evaluation. The average permeate flux in case of Hollow Fiber-43 to achieve concentration factor *(CF)* = 2 was 31.52 $l \cdot h^{-1} \cdot m^{-2}$ and in case of Hollow Fiber-60 to obtain *CF* = 4 from *CF* = 2 concentrate was 23.19 $l \cdot h^{-1} \cdot m^{-2}$. The average flux to reach *CF* = 4 in case of Carbosep and Ceraver modules was 27.75 and 86.13 $l \cdot h^{-1} \cdot m^{-2}$, respectively. The retention of protein by all types of membranes at *CF* = 4 was the same. The energy consumption in the case of Hollow Fiber-43 and -60, Carbosep and Ceraver to obtain *CF* = 4 was 3.14, 16.78 and 7.12 kWh/m^3 permeate collected. The lower energy consumed by Hollow Fiber modules was due to the lower operating velocity, *ie* 0.6 m/s as compared to 4.7 m/s in 1 the other 2 modules. The Ceraver microfiltration module P-19-40 was found to be the best for making quarg by ultrafiltration of coagulated skim milk of all the modules investigated. Although this module has a double energy requirement as compared to Hollow Fiber modules, its average flux is much higher. This module is easy to clean. On the other hand, Hollow Fiber membranes fouled by coagulated skim milk require vigorous cleaning with additional energy expenditure.

quarg / performance / ultrafiltration / coagulated skim milk / polymeric and mineral membranes

Résumé — Fabrication de quarg par ultrafiltration : comparaison des performances de modules à membranes polymères et à membranes minérales. *Les performances des modules d'ultrafiltration Hollow Fiber (HF43 et 60), Carbosep S7 et Ceraver (P-19-40) ont été comparées sur lait écrémé coagulé à pH 4,6 en vue de la fabrication de quarg. Les paramètres choisis ont été : le flux de perméation, la consommation d'énergie par* m^3 *de perméat et les coefficients de rétention en matière sèche totale, protéines, lactose et calcium. Le flux moyen de perméat pour le module HF43 pour atteindre la concentration x 2 était de 31,5* $l \cdot h^{-1} \cdot m^{-2}$ *et pour le module HF60, pour passer de la*

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concentration x 2 à la concentration x 4, de 23,29 $l \cdot h^{-1} \cdot m^{-2}$. Le flux moyen pour atteindre la concentration x 4 était, pour les modules Carbosep et Ceraver, de 27,7 et 86,1 $l \cdot h^{-1} \cdot m^{-2}$ respectivement. La rétention de protéines était la même pour tous les types de modules à la concentration x 4. La consommation d'énergie pour les modules HF43 ou HF60, Carbosep et Ceraver pour obtenir une concentration x 4 était respectivement de 3,14, 16,78 et 7,12 kWh par m^3 de perméat. La plus faible consommation d'énergie des modules HF est due à une plus faible vitesse de recirculation : 0,6 m/s contre 4,7 m/s pour les autres modules. De tous les modules testés, le module de microfiltration Ceraver P-19-40 s'est montré le meilleur pour la fabrication de quarg par ultrafiltration de lait écrémé coagulé. Bien que ce module consomme le double d'énergie par rapport aux modules Hollow Fiber, son flux moyen est bien plus élevé. De plus, il est simple à nettoyer. Par ailleurs, les membranes Hollow Fiber colmatées par le lait écrémé coagulé nécessitent un nettoyage intense qui entraîne donc une consommation d'énergie supplémentaire.

quarg / ultrafiltration / lait écrémé / coagulum / membrane minérale / membrane polymère

INTRODUCTION

Quarg is an unripened fresh cheese made from skim milk with high moisture content. It is milky white in colour and has soft body and texture with good spreadability. It has a clean but mildly acidic flavour (Winwood, 1983).

The consumer demand for quarg and other fresh cheeses is growing. The per capita consumption of quarg and other fresh cheeses in West Germany has attained 6.6 kg out of a total cheese consumption of 15.1 kg (Anonymous, 1987). The demand for this product is estimated to increase in the future (Kurmann, 1986).

Quarg is very popular throughout Europe; however, its availability in Canada and the USA is very limited. Sohal *et al* (1988) conducted a survey on the acceptance of quarg by Canadian consumers. They found that 77% of the respondents had not consumed quarg before in any form; however, a majority of them indicated an interest in purchasing the product. This shows a potential demand for quarg in Canada and also in the USA. The characteristics, manufacture, composition, future consumption trends in the USA and Europe of quarg have been reviewed (Jelen and Renz-Schaugen, 1989).

Because of the potential quarg market, considerable efforts have been made in the recent past to increase quarg yield while maintaining the best possible organoleptic and microbiological quality (Winwood, 1983). Some modified methods such as centriwhey, lactal, thermoquarg and ultrafiltration (UF) have been briefly described in the literature (Hayes, 1987). To produce 1 kg quarg by ultrafiltration, 3.6 kg of skim milk is required compared to 4.7 kg by the traditional method (Winwood, 1983). This is the lowest amount of skim milk per kg quarg production among all the available methods. Wietbrauk and Krell (1988) calculated the cost of quarg production (per kg) for different methods (traditional separator, thermoprocess, separator + whey proteins, and UF) of quarg manufacture and concluded that UF was the most economical (127.4 Pfennig/kg) method.

At present, there is a number of large ultrafiltration plants in operation for manufacturing quarg on a commercial scale in Germany and the United Kingdom (Herbertz, 1985; Darrington, 1987; Rockseisen, 1987). The quarg produced by UF is readily accepted by all consumers due to its smoother and creamier consistency and higher nutritive value. However, acid-type fresh cheese obtained by UF of milk at pH 6.6 contains too much calcium (Maubois *et al,* 1969) and often has a pronounced acidic bitter taste (Brulé *et al,* 1974, 1975). Nevertheless, this problem could be overcome by ultrafiltration of acid-coagulated (pH = 4.6) milk (Stenne, 1976; Mahaut *et al,* 1982).

In the present study, quarg was made by UF of coagulated skim milk at pH 4.6 (as suggested by Mahaut *et al,* 1982) with different modules in order to compare their performances. The objective of the study was to select the most appropriate module for the manufacture of quarg on the basis of permeate flux, energy consumption and retention of milk components.

MATERIALS AND METHODS

Raw milk

Raw cow milk obtained from the research center's farm was skimmed and heated at 95 °C for 5 min in a double-jacketed vertical vat under constant slow agitation.

Quarg cheese-making

The quarg cheese-making process is shown in figure 1. For each trial, 50 l skim milk cooled at 21–22 °C was inoculated with 0.1–0.2% conventional mixed starter culture of *Lactococcus lactis* subsp *lactis, cremoris* and *diacetylactis* and incubated for 16–18 h to obtain a pH = 4.6. The coagulated milk was heated at 60 °C for 5 min and concentrated 4 times at 50 ± 2 °C on 3 different UF modules (table I). With the Hollow Fiber type module, the concentration factor *(CF)* was 2 (12–13% on total solids (TS) on module HF 43 (fiber diameter = 1.1 mm), then from *CF* = 2 to *CF* = 4 (17–18.5% DM) on module HF = 60 (fiber diameter = 1.53 mm); the inlet (pi) and outlet (po) pressures were respectively 200 and 50 kP_{CL}; however they were 450 kPa (Pi) and 350 kPa (po) for mineral membranes (Carbosep and Ceraver). Dynamic pressure difference (Δ_p) during UF process for Hollow Fiber, Carbosep and Ceraver modules was 139, 88 and 91 kPa respectively.

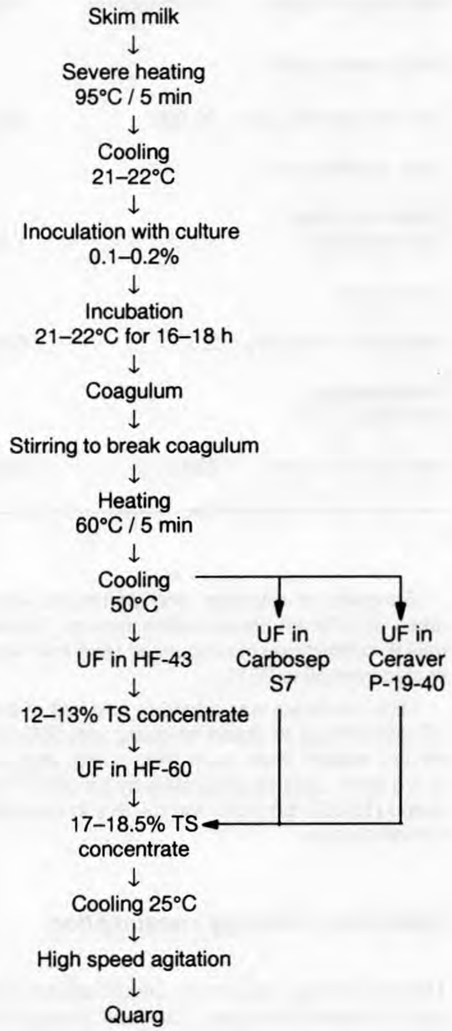

Fig 1. Process flow chart.
Diagramme de fabrication.

Table I. Specifications of different modules.
Caractéristiques des modules utilisés.

	Hollow Fiber-43	*Hollow Fiber-60*	*Carbosep S7*	*Ceraver P-19-40*
Manufacturer	Romicon, USA	Romicon, USA	SFEC, France	Ceraver, France
Membrane material	Polysulphone	Polysulphone	Metal oxide	δ-Al_2O_3
Membrane support	–	–	Sintered carbon	δ-Al_2O_3
Cut-off capability (Da)	50 000	50 000	50 000	-
Pore diameter (μm)	–	–	–	0.2
Fiber/tube inside diameter (mm)	1.1	1.53	6	4
No of tubes	–	–	7	19
Membrane area (m^2)	2.5	0.56	0.15	0.2
Pressure drop limit (kPa)	–	–	400	400
Pressure limit (kPa)	238	238	800	800

Samples of retentate and permeate were taken at different concentration factors. The final UF concentrate of coagulated skim milk was cooled from 50 to 25 °C.

High fat cream was added to one half of the UF concentrate to obtain fat quarg with 50% fat on dry matter, then skim milk quarg and fat quarg were agitated separately by an ultra-high speed (10 000 rpm) rotor for 15–25 s to obtain a smooth texture.

Estimation of energy consumption

The total energy required for ultrafiltration is the sum of several energies. The total energy *(E)* required during ultrafiltration can be expressed as:

$$E = E_T + E_P + E_Q$$

where E_T is the thermal energy required to maintain the process fluid temperature. The temperature of the milk in the experiment was maintained through a tubular heat exchanger. However, the thermal energy was not taken into account in our energy estimations.

E_P is the energy required for maintaining transmembrane pressure and E_Q is the energy needed by the recirculating pump to maintain fluid velocity throughout the module. In ultrafiltration applications, E_P is always very low as compared to E_Q. Therefore, E_P was not taken into account as a first approximation in estimating energy consumption of the ultrafiltration module. Only E_Q was calculated from the power P (W) required to maintain the circulation;

$$P = \Delta_p \cdot Q \text{ (W)}$$

where Δ_p(Pa) is the pressure drop along the module and Q (m^3/s) is the average flow rate through the module during the operation measured by a flow meter. E_Q in kWh was calculated by measuring the power P at regular short intervals and integration thereof;

$dE_Q = \Delta_p \cdot Q \cdot d_t$ (kWh)

$E_Q = \int \Delta_p \cdot Q \cdot d_t$ (kWh)

E_Q was expressed on the basis of permeate during the entire ultrafiltration time (m^3 perm) in terms of kWh/m^3 perm.

Analytical methods

The samples were analysed for total solids (TS) by gravimetric procedure. The protein content was taken as 6.38 times the nitrogen content determined by Kjeldahl analysis. Calcium and lactose were determined by a Technicon autoanalyzer. The pH was measured using a pH meter.

Sensory evaluation

Skim milk quarg and fat quarg were judged by a panel of judges on the 9-point hedonic scale ranging from 9 (liked extremely) to 1 (disliked extremely).

RESULTS AND DISCUSSION

The performance of different modules for ultrafiltration of coagulated skim milk for making quarg was compared by considering 3 basic parameters, *ie* permeate flux ($l \cdot h^{-1} \cdot m^{-2}$); energy consumption (kWh/m^3) and retention coefficients ($1 - C_P/C_r$) at $CF = 4$, where C_P and C_r were constituents content in permeate and retentate respectively.

Permeate flux

As shown in figure 2, the permeate flux decreased very rapidly up to $CF = 2$, then very slowly from 2 to 4 for different types of membrane. The flux decrease was caused by the deposited layer above the membrane composed of a selective concentration of caseins and soluble protein particles (Mahaut *et al*, 1982; Bennasar and Tarodo de la Fuente, 1987). The permeate flux drop between $CF = 1$ to $CF = 2$ was higher for Hollow Fiber (70%) compared to 65% for Carbosep and 46.5% for Ceraver.

The lower permeate flux drop and the higher permeate flux for Ceraver membrane were probably due to higher asymmetry and to larger membrane pore size (0.2 μm).

Energy consumption

Energy required in kWh/m^3 permeate collected for obtaining $CF = 4$ for different modules is given in table II. It was lowest for Hollow Fiber; and 2.3 and 5 times higher for Carbosep and Ceraver respectively than that of Hollow Fiber.

Hollow Fiber consumed the lowest energy per m^3 permeate collected and Carbosep consumed the highest. The low energy requirement of the Hollow Fiber module was due to its lower average recirculating velocity of 0.6 m/s compared to 4.7 m/s of the other 2 modules.

The lower energy consumption per m^3 permeate by Ceraver microfiltration module as compared to Carbosep was essentially due to its significantly higher average permeate flux to obtain $CF = 4$.

Retention coefficient

Average retention coefficients of total solids (TS), protein, lactose and calcium at $CF = 4$ are shown in table II. Retention coefficient of protein by different membranes was practically the same (98%).

Table II. Mean permeate flux, energy consumption and retention coefficient with different modules.
Débit moyen de perméat, consommation d'énergie et coefficient de rétention des différents modules.

Sr No	*Type of module*	*No of trials*	*Operating trans-membrane (kPa)*	*Mean permeate flux to obtain CF = 4 l•h⁻¹•m⁻²*		*Mean energy consumption to obtain CF = 4 (kWh/m³ permeate)*	*Retention coefficient at CF = 4*			
				HF-43 up to CF = 2	*HF-60 from CF = 2 to CF = 4*		*Total solids*	*Protein*	*Calcium*	*Lactose*
1	Hollow Fiber HF-43 and 60	4	125	31.52	23.19	3.14	0.765	0.982	0.086	0.051
2	Carbosep S7	3	400	27.75		16.78	0.720	0.978	0.094	0.116
3	Ceraver micro-filtration P-19-40	4	400	86.13		7.12	0.729	0.979	0.194	0.017

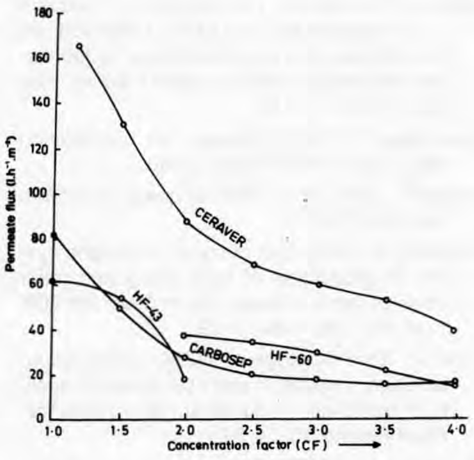

Fig 2. Evolution of permeation flux as a function of concentration factor with different UF-modules at 50 ± 2°C.
Évolution du flux de perméation en fonction du facteur de concentration avec les différents modules UF à 50 ± 2°C.

Calcium retention coefficient was 2-fold higher in Ceraver module due to the lower permeability of the adsorbed layer to soluble calcium. The role of the adsorbed layer in controlling the permeability of the principal membrane has been confirmed by Vetier *et al* (1988) and Attia *et al* (1991a) through their electron microscopic and electrophoretic analysis of the adsorbed layer on the microfiltration (0.2 μm) alumina membrane. The formation of the adsorbed layer and its characteristics are also affected by the interaction between deposit and membrane material, and membrane pore size in addition to pH of milk (Vetier *et al,* 1986; Attia *et al,* 1991b).

The lowest retention of lactose by Ceraver membrane (1.7%) is also due to the higher permeability to lactose of the adsorbed layer.

Sensory evaluation of quarg

The average sensory evaluation scores on a 9-point hedonic scale are shown in table III. Average sensory evaluation scores for traditional skimmed milk quarg (market sample), UF-skimmed milk quarg and UF-fat quarg (fat 50% on dry matter) were 8.0, 6.2 and 7.5, respectively. The UF skimmed milk quarg was given low scores because of its less acid taste at pH 4.6. This may be due to the buffering effect of whey proteins. Otherwise, UF-quarg was smoother, creamier and more spreadable as compared to traditional quarg. UF-fat quarg was better appreciated than the UF-skimmed milk quarg.

Table III. Sensory evaluation scores of different types of quarg.
Évaluation sensorielle des différents types de quarg.

Type of quarg	*Scores*	*Remarks*
Traditional skimmed milk quarg (market sample)	8.0	Good mild acidic flavour, creamy and plastic consistency and dense body
UF-skimmed milk quarg	6.2	Good flavour, slightly less acidic, smooth texture, loose body, spreadable
UF-fat quarg (fat 50% DM)	7.5	Characteristic full flavour, smooth and creamy consistency, loose body, spreadable

CONCLUSION

A fairly good quality quarg with mild acidic flavour, smooth and creamy texture and of higher spreadability can be made by ultrafiltration. UF-quarg is most suited as a spread.

On the basis of flux and retention coefficient data, it can be concluded that the Ceraver microfiltration module P-19-40 is the best of the modules investigated for making quarg from coagulated skim milk.

Although the energy requirement for making quarg of Hollow Fiber modules is lowest, they also have the lowest permeate flux. These modules have the drawback of severe fouling with coagulated skim milk. The Hollow Fiber membranes fouled during ultrafiltration of coagulated skim milk were very difficult to clean to regain initial water flux. The vigorous cleaning procedure required very high energy and also affected membrane life and thereby replacement cost.

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