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RECENT AND EMERGING APPLICATIONS OF MEMBRANE PROCESSES IN THE FOOD AND DAIRY INDUSTRY

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Membrane processes have been major tools in food processing for more than 25 years. The food industry represents a significant part of the turnover of the membrane manufacturing industry world-wide. The main applications of membrane operations are in the dairy industry (whey protein concentration, milk protein standardization, etc.), followed by beverages (wine, beer, fruit juices, etc.) and egg products. Among the very numerous applications on an industrial scale, a few of the main separations which represent the latest advances in food processing, are reported. Clarification of fruit, vegetable and sugar juices by microfiltration or ultrafiltration allows the flow sheets to be simplified or the processes made cleaner and the final product quality improved. Enzymatic hydrolysis combined with selective ultrafiltration can produce beverages from vegetable proteins. In the beer industry, recovery of maturation and fermentation tank bottoms is already applied at industrial scale. During the last decade significant progress has been made with microfiltration membranes in rough beer clarification which is the most important challenge of this technology. In the wine industry the cascade cross-flow microfiltration (0.2 μm pore diameter) – electrodialysis allows limpidity, microbiological and tartaric stability to be ensured. In the milk and dairy industry, bacteria removal and milk globular fat fractionation using cross-flow microfiltration for the production of drinking milk and cheese milk are reported. Cross-flow microfiltration (0.1 μm) makes it possible to achieve the separation of skim milk micellar casein and soluble proteins. Both streams are given high added value in cheese making (retentate) through fractionation and isolation of soluble proteins (β -lactoglobulin; α -lactalbumin) (permeate). At last, a large field of applications is emerging for the treatment of individual process streams at source for water and technical fluids re-use, and end-of-pipe treatment of wastewater's, while reducing sludge production and improving the final purified water quality.

Keywords: food industry; milk; dairy; wine; beer; vegetable protein; beverage; waste stream; membrane process

INTRODUCTION

Membrane processes have become major tools in the food processing industry over the last 25 years. The food industry represents 20 to 30% of the current-€250 million turnover of membranes used in the manufacturing industry worldwide. The growth in this market is around 7.5% per year. Several hundreds of thousand square meters of membrane (Ultrafiltration, UF: 400,000; Nanofiltration, NF: 300,000; Reverse osmosis, RO: 100,000; Microfiltration, MF: 50,000) are currently operating. The main applications of membranes are in the dairy industry (close to 40%, of which over 10% are used for milk protein standardization), followed by

beverages (wine, beer, fruit juices, etc.) and egg products (2%). Other fields are emerging: fruit and vegetable juices and concentrates, waste streams, co-products (recovery and recycling of blood plasma in abattoirs) and technical fluids (brines, cleaning-in-place solutions).

Among the very numerous industrial applications, a few outstanding processes represent the very latest advances in food processing. They have been selected to show the trends that are emerging in the treatment and transformation of raw products from agriculture to safe food products or beverages well accepted or required by the consumer. The objective of the present paper is to complete and up-date the overall information reported recently by Cheryan¹ and Daufin *et al.*².

INTEGRATION OF MEMBRANE SEPARATIONS IN FOOD PROCESSING

The main features of membrane separations adopted by the food industry are:

- In the preparation of traditional food products, they contribute to simplification of flow sheets (replacement of two or more steps) and to the improvement of process performance (clarification, etc.) and food quality (low temperature operation, etc.).
- Often, they participate in innovative processes and/or products. In effect, in the last decade food requirements have evolved with the triple key words: Nutrition + Dietetics + Health. This evolution makes it necessary to design novel foods and intermediate food products by manufacturing fractions and co-fractions from initial products, which must comply with the requirements of the consumer: Safety + Novelty + Diversity + Quality. In many cases totally novel food products (cheeses, etc.) have been manufactured using membrane separation.
- With respect to the environment, membrane separations are regarded as clean processes: a substitute for the use of polluting materials (diatomaceous earth in clarification of wine, beer, fruit juices, etc.); usually co-fractions can be given added value (fractionation of protein containing fluids, etc.); very well adapted to the treatment of effluents (evaporation condensates, ultrafiltrates, nanofiltrates, osmosates, technical fluids like brines and cleaning-in-place solutions), and wastewater's.
- Compared to competitive concentration (thermal processes) and separation operations (decantation, filtration, centrifugation, chromatography, etc.) membrane processes are attractive to industry, wherever they can be used, since they are easy to implement, have great flexibility (module systems), are compact (more or less depending on the type of module: spiral-wound, hollow-fiber, plate-and-frame, tubular), good automation. Membrane processes give the food industry three advantages: food safety, competitiveness, environmental friendliness.

BEVERAGES

According to Cheryan¹, Decloux & Prothon³, and Girard and Fukumoto⁴ membrane operations are most often combined with each other (successive filtrations with different molecular weight cut-off membranes) or with other types of separation (pre-treatment by enzyme, depectinization or flocculation; concentration by evaporation; ion exchange for demineralization, de-acidification, discolouring, etc.) in the fruit, vegetable and sugar juices field.

Fruit and Vegetables Juices

In the field of fruit juices, the major application of membranes concerns the clarification by MF or UF. The apple juice industry has taken advantage of the potential of membranes due to the requirement of limpidity and a large production that justifies long-term investment. The few known industrial plants run with average permeation flux around $100 \text{ l h}^{-1} \text{ m}^{-2}$ with optimal operating parameters: previous depectinization (enzyme); 50°C ; transmembrane

pressure, $\Delta P = 0.5\text{--}5 \text{ bar}$ and cross-flow velocity: $3\text{--}7 \text{ ms}^{-1}$ depending on the type and characteristics of the membrane. Cider is more complex to treat compared to apple juice, since it contains yeasts and fermentation products (alcohol, CO_2). For citrus (orange, lemon, grapefruit, tangerine) and pulpy fruits, membrane filtration allows pulp (retentate) and serum (permeate) to be separated. They are treated on separate lines (de-acidification, concentration, pasteurization, etc.) before their eventual final mixing. Industrial UF for the clarification of orange juice is limited by fouling⁵. On the other hand, industrial UF of lemon juice⁶ meets an increasing need of limpid concentrated juices for use either as acidulous or bitter or astringent agent in beverages, sauces or domestic cleaners.

Novel processes also combine membranes from MF to RO to allow the production of fruit juice concentrates in which the organoleptic quality after dilution is close to fresh fruit drink. The freshnote process allows dry solid (DS) concentrations over $60 \text{ g DS } 100 \text{ g}^{-1}$ to be obtained using an UF step (pulp/filtrate) and RO with high retention in the first 2 stages and low retention in the last 2 stages⁷. MF and RO or osmotic evaporation combined with previous enzymatic treatment of raw material, can produce either clarified concentrates from turbid juices or pulps (lime, lemon) or stable pulps concentrates, with a high sugar content (65%) and suspended solids (30%) from fruit pulps (mango)⁸⁻¹⁰. This technology which differs from the classical concentration process allows the manufacture of stable clarified concentrates of pulps, difficult to prepare any other way. Moreover, the technological characteristics of these products, which provide the starting material for the industry manufacturing fruit juices and beverages and other sectors of the food industry, are attractive: products dilutable with stable or no turbidity, pastry products with high dry matter in pulp, sugar, organic acids, colouring agents, aromas. These membrane operations help with the creation of novel products and keep organoleptic properties of intermediate products reasonably good for export or of final products for domestic consumption in developing countries. MF and UF or RO are reported for the clarification or concentration of exotic (pineapple, kiwi, etc.), pulpal (apricot, peach, pear) and red (strawberry, blackcurrant, raspberry, cherry) fruits. The concentration of tomato pulp of high organoleptic quality by UF¹¹ and by RO (4.5 to 9% DS) are the major industrial membrane processes applied to vegetable juices. Other vegetables (cucumber, carrot, mushroom, celery, etc.) are clarified and concentrated by UF and RO¹² and even demineralized by NF (NO_3^- , NO_2^- , etc.) which stabilizes the concentrate of red beetroot, amongst others¹³.

Sugar Juices

Numerous beverages are formulated by incorporating liquid sugar or glucose syrups. Clarification of these ingredients by MF or UF is an industrial reality with very rapid development where ceramic membranes give higher filtration flux¹⁴. The advantages of membrane operations over traditional purification are: better removal of macromolecules, microorganisms, and compounds that participate in fouling evaporator tubes and improved quality of final product. Combination of membrane filtration and other types of separation (pre-treatment by flocculation,

chromatography, discolouration or demineralization with ion exchange membranes or NF) are expected to yield large modifications of flow sheets. Liquid sugar or glucose syrup used for the manufacture of sweetened beverages requires high standards: turbidity $< 5 \text{ IU}^{15}$, colour $< 45 \text{ ICUMSA}$, ash $< 0.05\% \text{ DS}$. Three steps, clarification, discolouration and demineralization are included in the flow sheet. Clarification by MF (200–20 nm) has optimal performance (DS flux) at $30 \text{ g DS } 100 \text{ g}^{-1}$ and is competitive compared to traditional filtration with additives for starch hydrolysate, but the cost is too high for remelted sucrose.

Cane Sugar Mill

The first industrial ultrafiltration plant for clarified cane juice was started in 1994¹⁶. The process conditions 940 m^2 membrane area 300 t h^{-1} of juice at $16 \text{ g DS } 100 \text{ g}^{-1}$ and 98°C temperature. The final volume reduction ratio (VRR) is 10 and the retentate is recycled to the clarification plant. This extra UF step allows the manufacture of less coloured sugar and improves the sugar extraction yield by treating clear molasses with chromatography.

Cane Sugar Refinery

At a cane sugar refinery a discolouration step is compulsory. The use of anionic resins (chloride) is one of the techniques used, but it yields polluting eluates (coloured brine). The effluent is composed of over 90% of the salt not consumed after regenerating the resin. Three industrial plants are operating using NF of these eluates¹⁷ to separate colourings and salts. Using VRR salt and water consumption can be reduced by 50 to 80% and the volume of effluents is reduced by 3 to 10. The combination micro-filtration-flocculation is applied in one industrial plant¹⁸ and yields a discolouration rate and permeation flux which are satisfactory from an economical point of view.

Manufacture of Glucose Syrups From Wheat Starch: Operating an Industrial UF Plant

The industrial plant in operation since 1992, produces glucose syrups which can be used as food ingredients for biscuits, soft drinks, ice creams, candies, jams, fruit preserves and as carbon sources for fermentations¹⁹. Ultrafiltrations (300 kDa weight cut-off ceramic membranes) are used to eliminate the suspended solids and the proteins from wheat hydrolysates at the flow-rate of $50 \text{ m}^{-3} \text{ h}$. The dextrose equivalent of the filtered sugar is not affected. The continuous three-stage filtration unit of 570 m^2 membrane area is composed of six identical lines: five are in production while the sixth is in cleaning²⁰. The retentate at $\text{VRR} = 20$ of the manufacturing lines, is diafiltered in a 57 m^2 membrane unit in order to reduce the loss in sugar down to less than 0.5%. The average flux at 70°C is $100 \text{ l h}^{-1} \text{ m}^{-2}$.

Vegetable Proteins

Vegetable proteins and their derivatives are commonly used for the manufacture of a variety of beverages²¹.

- The extracts of soybean (tonyu or soymilk) contain proteins (47%), lipids (24%) and glucides (15%). UF

and diafiltration at high temperature ($65\text{--}85^\circ\text{C}$), with a multistage plant can yield a protein (47–60%) and lipid (24–34%) enriched concentrated tonyu (12 to 24% dry matter). The four major advantages of the membrane process are:

- reduction of transport costs of a crude tonyu from production factory to the flavouring and bottling unit if they belong to different companies;
 - lowering the flatulent alphagalactoside content;
 - better manufacture of an intermediate product for food industry (ice creams; functional properties challenging that of soybean isolates despite its higher cost);
 - more unctuous, more viscous tonyu with a smooth structure. Provided that the tonyu is concentrated enough, it can be salt-coagulated by a continuous process, giving a standardized tofu²².
- Beverages for sportsmen with protein hydrolysates and glucides;
 - Beverages for parenteral nutrition or for people allergic to various proteins, including native proteins or hydrolysates.

Compared to traditional processes, membranes make it possible to standardize concentrations of grain extracts and to decrease concentration of certain undesirable compounds. They display several advantages: increased productivity; better mastering of hydrolysis; limited lack of proteins, for use in allergenic products; desalination of the hydrolysate and eventually fractionating it for technological (functional properties of some peptides) or organoleptical reasons (bitter hydrophobic peptides). Peptides cannot be fractionated by size only, as small peptides remain in the retentate²³. Conversely, by using a combination of membranes and physico-chemical parameters, differences of electrical charges or hydrophobicity of peptides and membranes can be successfully exploited. For example, wheat gluten hydrolysates can be fractionated by UF at acidic pH with ZrO_2 -coated membranes in a retentate containing 80% of hydrophobic and positively charged peptides and a permeate containing 80% of hydrophilic and neutral peptides²⁴.

The retentate of the manufacturing lines is diafiltered in a 57 m^2 membrane unit in order to reduce the loss in sugar down to less than 0.5%.

BEER

In the food industry, the brewing sector holds a strategic economic position, with world beer production exceeding 1.35 billion hectolitres per year in 1999²⁵. Beer is the second most consumed beverage in the world behind tea, and it continues to be a popular drink. This market hides an important heterogeneity of production capacity^{26–27}; for example the world's 10 largest brewing groups share almost 50% of the world production (production capacity superior to $100 \text{ million hl year}^{-1}$ for Anheuser-Bush and Interbrew groups). In contrast, a microbrewery may start its activity with an annual production close to 1000 hl^{28} . The brewing industry has an ancient tradition and is still a dynamic sector open to modern technology and scientific progress. A lot of work is being done to maintain or increase brightness and clarity, important quality

factors^{29–30}. Filtration removes solids and particles from malt, hops and yeast³¹. During production, beer alternately goes through three chemical and biochemical reactions (mashing, boiling, fermentation and maturation) and three solid–liquid separations (wort separation, wort clarification and rough beer clarification)³².

This overview of the brewing process offers potential applications for MF³³. The solid–liquid separation constitutes a real practical problem in brewing. The conventional dead-end filtration with filter-aids, such as diatomaceous earth (Kieselguhr) has been the standard industrial practice for more than 100 years and will be increasingly scrutinized from economic, environmental and technical standpoints in the coming century^{34–36}. Water consumption (6.5 hl hl⁻¹ of beer in average with minima: 3.4 and maximum: 13.3) needs to be reduced to maintain competitiveness and with respect to environmental legislation³⁷. Consideration of the brewing process indicate two areas where MF might play a useful role: (i) *loss reduction* in the brewing process, (ii) *as a technological alternative* to the conventional solid–liquid separations. In the following paragraphs, the authors develop the aim of each specific operation and present a statement of the major scientific works and industrial applications dedicated to brewing over the last 5 years (1995–2000).

Loss Reduction

Loss reduction concerns two applications: the recovery of extract during the wort clarification and beer recovery from tank bottoms (fermentation and maturation vessels). At present, tank bottom recovery constitutes the principal membrane application in brewing.

Extract recovery from loose trub suspension

The classification and taxation of beers are determined from the original and specific gravities. These factors measured in the clarified beer are functions of the extracted soluble matters during mashing. A beer could be down-graded if the loss of extract is too great, which means that the recovery of this extract is of interest. It is also considered that if the extract is initially recovered, it might lead to a better quality integrated recovery procedure³⁸. Extract recovery after trub separation with MF may render the following possible: the production of a permeate with acceptable quality characteristics (original gravity, bitterness, colour, total fatty acids); a substantial volume reduction of trub (over 50%); the possibility of operating at 75°C; the achievement of an economic flux.

However, no scientific or industrial practice has been reported which achieves extract recovery through cross-flow filtration over last decade. This operation remains a potential application of cross-flow microfiltration but is not a priority.

Green beer recovery fermentation tank bottoms

Low fermentation beers are produced through two fermentation steps, the primary fermentation is stopped when 90% of the fermentable matter is consumed. A rapid cooling of the tank stops this fermentation and causes the flocculation of insoluble particles and the sedimentation of yeast. The tank bottom becomes full of yeast and ‘green

beer’. MF of fermentation tank bottoms may allow beer loss to be limited. At present, the fermentation tank bottom generates a beer loss of around 1–2% of production. The permeate recovered by MF could be recycled in the wort or in the maturation vessels^{39–40}.

Rough beer recovery from maturation tank bottoms

In brewing, yeast surplus is recovered by natural sedimentation at the end of the second fermentation and maturation. Commercial sale of this surplus ($\approx 5\text{€}/\text{ton}$) can be made to the animal feed industry³⁶. This brewing by-product has a dry matter content close to 10% w/w. The application of MF could reduce beer losses (or waste) which represent between 1.5 and 3% of the total volume of produced beer. The beer recovered may be returned to the maturation vessel or sent to the final clarification. However, the different composition of the tank bottom beer may prevent a direct dilution into the rough beer before filtration^{41–43}.

Two fundamental differences exist among tank bottoms: (i) the fermentation vessels have high yeast cell content and high viscosity; (ii) the maturation vessels have high protein and polyphenol contents, and fewer yeast cells, and are characterized by low viscosity (close to that of beer). In order to recover ‘green beer’ and ‘rough beer’ from tank bottoms, natural sedimentation, centrifugation and a filter-press may be used. However, centrifugation is expensive and may damage the permeate quality because of yeast cell degradation. Filter-presses provide a relatively low moisture solid discharge and consequently high extract recovery. Sufficient clarification of the filtrate is not obtained. The use of MF aims:

- (i) to produce a permeate of acceptable quality including flavour and haze, with minimal loss of original gravity, colour and bitterness;
- (ii) to process a retentate of between 2–4% dry weight to a minimum of 20% solids;
- (iii) to operate at low temperatures (close to 0°C);
- (iv) to achieve economic flux and hygienic beer recovery.

Almost, all of the membrane area installed in breweries around the world is dedicated to the recovery of beer from fermentation and maturation tank bottoms. At present, this membrane application almost constitutes an industrial standard. The problem today is more a of commercialization than a food engineering science. Since 1994 numerous industrial applications^{44–49} have been reported in addition to scientific papers^{50–55}. MF enables a 20–30% w/w concentration to be reached and several industrial units already use it. More than 50–60% of the yeast sediment is recovered as a high quality beer (equivalent to a volume reduction ratio between 2 and 3). The membrane filtration becomes competitive in comparison to the filter-press for waste reduction. The recovered permeate recycled in the brewing process, at a rate of 2 to 5%, allows beer loss and costs to be reduced. Various systems are in use and it has been shown that ceramic (0.4–0.8 μm ⁴⁴) or polysulfone (0.6 μm ⁵¹) membranes concentrate solids from 12–15% to 20–22%. The payback is less than 2 years, regarding the recovery of sterile beer from yeast beer, with 0.4–0.8 μm pore diameter multi-channel ceramic membranes installed in 1 million hectolitres capacity breweries. Bock and Oechsle⁴⁷ explained that brewing plants are running with ceramic

membrane made of α -aluminium oxide (multi-channel membrane: 19 channels, length: 1020 mm, mean pore diameter: $0.80\ \mu\text{m}$). Surplus yeast can be processed with about $17\text{--}20\ \text{lh}^{-1}\ \text{m}^{-2}$ up to a concentration of 20% w/w (transmembrane pressure up to 3 bar), three process options exist—batch, semi-batch, continuous. This material can be cleaned in place since it is resistant to caustic, acid and oxidizing sterilants even at high temperature (above 90°C). Snyder and Haughney⁵⁴ describe a new system called VMF (Vibrating Membrane Filtration) produced by PallSep. The system differs from traditional cross-flow filtration systems in that the shear at the membrane surface is generated mechanically (by vibrational energy, 60 Hz) and not from high cross flow rates. Recovery of beer from surplus yeast could be achieved with a filter disc of PTFE membrane with the same performances as for the other processes.

Technical Alternative

MF is aimed at three applications: mash separation, clarification of rough beer, cold-sterilization of clarified beer before conditioning. Industrial applications concern, essentially, the clarification of rough beer and sterile filtration of clarified beer.

Mash separation

The mashing process is one of the initial operations in the brewery and has to solubilize the content of malt and cereal grain in water. After extraction, the spent grains and wort (water with solubilized extracted matter) are called mash and need to be separated^{56–57}. MF can be applied to the separation of wort and it should:

- (i) produce a high quality sweet wort;
- (ii) complete the operation quickly and with economical flux;
- (iii) concentrate the initial amount of solid in the mash (typically 25–30%) up to a maximum value;
- (iv) extract more than 90% of the spent grains from wort.

The only pre-industrial application regarding the separation of wort from mash was built by APV (US Patent 4844932, 1989). The amount of solids present in the mash (20–30%) is so high that complete separation of wort using a single stage cross-flow filter could only be achieved by accepting low flux. A two-stage process is recommended. Daoud⁵⁶ used tubular stainless steel membranes (pore size: $70\text{--}80\ \mu\text{m}$) in the first stage and reported flux in excess of $1000\ \text{lh}^{-1}\ \text{m}^{-2}$ with 90 to 95% removal of solid.

Bühler *et al.*⁵⁸ reported that cross-flow membranes system could be applied to the separation of wort. Mash separation is achieved in a two-stage process. The first stage separates the spent grains and all particles bigger than $15\ \mu\text{m}$ using a centrifuge decanter. A second clarification using $1.3\ \mu\text{m}$ cross-flow ceramic membranes is then possible. Flux obtained is about $100\ \text{lh}^{-1}\ \text{m}^{-2}$ (at 1.3 bar transmembrane pressure over 1 hour) within quality specifications.

Clarification of rough beer

Beer clarification is probably one of the most important operations. Rough beer is filtered in order to eliminate yeast and colloidal particles responsible for haze; in addition, this

operation should also ensure the biological stability of the beer. This operation should comply with the haze specification of a lager beer in order to produce a clear bright beer. Standard filtration consists of the retention of solid particles (yeast cells, macro-colloids, suspended matter) and solutes responsible for haze. The variety of compounds (chemical diversity, large size range) to be retained, makes this operation one of the most important in beer production and also one of the most difficult to control. The use of MF is to provide an alternative to the conventional dead-end filtration with filter-aids such as diatomaceous earth. However, this operation should satisfy the same economic and qualitative criteria^{43–59}. MF should be able to produce a clear and bright beer with similar quality to a Kieselguhr filtered beer, to perform a separation in a single-step without additives to operate at low temperature (0°C), and to achieve economic flux.

Among the potential applications of cross-flow microfiltration, clarification of rough beer represents a large potential market (approximately $200,000\ \text{m}^2$ surface). Clarification of rough beer should be considered as an emerging market for membrane applications in the food industry. Industrial experiments however, encountered two main problems: (i) the control of fouling mechanisms, (ii) the enhancement of permeate quality. Since 1995, a lot of research has indicated the economic and scientific stakes of the clarification of rough beer. Recent scientific and industrial studies have dealt with (i) fouling mechanisms^{50,60–69}; (ii) relation between quantitative and qualitative performances^{50,60,70–71}; (iii) industrial applications^{46,72–73}. MF suffers from a low permeate flux in comparison to the conventional dead-end filtration with filter-aids such as diatomaceous earth (usual flux ranges from 100 to $500\ \text{lh}^{-1}\ \text{m}^{-2}$). However, the first industrial plants are running, for example, a MF unit of rough beer with a capacity of $10,000\ \text{l/h}$ ⁷² at Heineken. The plant contains 10 hollow fibre modules (pore size: $0.45\ \mu\text{m}$, length: 1 m, inner diameter: 1.5 mm, filter area: $9.3\ \text{m}^2$). The key of this process is a specific cleaning procedure combining an enzymatic step, an alkaline step and a strongly oxidative step which has been successful in maintaining run times of between 7 and 10 h for about 50 runs. Heineken and Norit Membrane Technology have patented this procedure. Filtration is processed at 0°C , $2\ \text{m}^{-1}\ \text{s}$ flow velocity and up to 1.6 bar transmembrane pressure. During filtration, 7 min periods of back flushing are applied hourly to remove the reversible fouling that has built up. The flux is maintained at $100\ \text{lh}^{-1}\ \text{m}^{-2}$ and clarified beer respects the European Brewery Convention (EBC) standard in terms of turbidity (close to 0.6 EBC unit), bitterness, total extract, colour, and protein content. The cost of membrane filtration for bright beer is about 0.68€/per hectolitre.

Cold sterilization of clarified beer

The clarification of rough beer is usually followed by pasteurization so as to ensure the microbiological stability and the conservation of beer. Currently, heat treatment is mainly performed using flash pasteurization before conditioning. Sterile filtration appears interesting and allows the elimination of the organoleptic problems induced by heat processing^{74–76}. MF will have to face several challenges: (i) to produce a microbial free beer without a negative change

in beer quality by operating at low temperature (close to 0°C); (ii) to ensure beer stability (biological, colloidal, colour, aroma and flavour, foam stability); (iii) to achieve economical flux. Provided it complies with these considerations MF would be an alternative to pasteurization and dead-end filtration with cartridges.

Cold-sterile filtered beer (draught beer or bottled beer) corresponds to a strong demand from consumers for quality and natural products. The objective of eliminating heat treatment of the finished product is achieved with membrane cartridge systems (dead-end filtration) installed directly upstream of the filling system^{77–81}. However, cold-sterilization by cross-flow membrane is under progress and could be realized^{66,82–83}.

In conclusion, cross-flow filtration in brewing offers several advantages, some of which are already in use:

- (1) CFMF is already an industrial standard for the treatment of fermentation and maturation tank bottoms;
- (2) CFMF constitutes an emerging application for the clarification of rough beer and cold-sterilization of clarified beer;
- (3) CFMF could be interesting in the recovery of extract from loose trub suspension and in the separation of wort from mash.

However the new processes should be compared to the technical, qualitative and economical requirements of the conventional industrial process relating to beer quality standards (reference to the European Brewery Convention norm, see Table 1). To be convinced of the interest of the brewing industry in cross-flow membrane processes, it is instructive to note those patents concerning brewing and filtration. Since 1994, more than 10 patents have been published in Europe. Their objectives were to improve conventional filtration^{84–86} or to protect new filtration process^{87–93}.

WINE: NOVEL PHYSICAL TECHNIQUES FOR WINEMAKING

Wines are made using a range of procedures with the objective of marketing clear and stable products. These

procedures generally aim at limiting the presence of micro-organisms, yeast and bacteria in wine and reducing the risks of tartar crystals and precipitates of colouring matter forming in the bottle. New technology substitutes traditional methods with the objective of simplifying operations and/or making them more selective or economic; it can also be the impetus for new products⁹⁴ MF and electro dialysis (ED), have been significantly developed on an industrial scale since 1997.

Cross-Flow Microfiltration for Microbiological Limpidity and Stability

MF can ensure microbiological limpidity and stability in a single operation. This has no effect on the quality of the wine treated and meets users' requirements in that it yields products with low turbidity (less than 1 NTU), low in germs ($0.2 \mu\text{m}$ average pore diameter) and it maintains the wines organoleptic qualities⁹⁵. Many applications are, henceforth, available where wine makers can treat their wine after fermentation.

MF is performed at room temperature with tubular (1.5 mm inner diameter) polysulfone membrane ($0.2 \mu\text{m}$ pore diameter), 2 m s^{-1} , cross-flow velocity, $1.2 \times 10^5 \text{ Pa}$ transmembrane pressure and periodical back-flushing (every two minutes). After the wine batch has been treated, rinsing and cleaning are automatically carried out. Long runs (10–20 h) can be operated with average permeation flux in the range 55 to $100 \text{ l h}^{-1} \text{ m}^{-2}$ depending on the type of wine (white, rosé, red, fortified red or must, etc.). For unfiltered wines, i.e. with high turbidity, flux is over $60 \text{ l h}^{-1} \text{ m}^{-2}$. For wines at final filtration, before bottling or preclarified, flux can be as high as $100 \text{ l h}^{-1} \text{ m}^{-2}$. The analytical characteristics of various wines—turbidity, filterability index, colour, retention of colloids, dissolved gas—are not significantly altered by MF as compared to control batches (Kieselghur, plates). Yeast and bacteria are reduced by a factor in the range 2–60 (yeast) to 5 – 10^4 (bacteria). With regard to sensory analysis, MF is the only technique which yields limpid wines and does not lead to 'thinning' or qualitative losses of the end product, as was stated after the first trials of MF in the early nineties. In spite of their high potential (chemical inertness, life time, thermal and mechanical resistance, easier cleanability) ceramic membranes are not yet well developed

Table 1. Average permeate flux and critical membrane cut-off versus the membrane applications in brewing.

Membrane filtration	Objective	Permeate flux $\text{l h}^{-1} \text{ m}^{-2}$	Critical pore size μm	Comments
MF of clarified beer	Cold-sterilization	15–60	0.20–0.50	Below $0.20 \mu\text{m}$, the essential beer compound retention is too high. The product is clear, bright and near sterile but becomes insipid (loss of colour, dry matter, bitterness and foam).
MF of rough beer	Rough beer clarification	25–45	0.50–0.80	Below $0.50 \mu\text{m}$, there is a loss of the essential compounds, dry matter and bitterness but the clarity and brightness are accurate. Above $0.80 \mu\text{m}$, the permeate is turbid because of a too weak retention of the components responsible for haze formation.
MF of fermentation and maturation tank bottoms	Green beer and rough beer recovery	10–50	1.0–2.0	Below 1.0 – $2.0 \mu\text{m}$, the permeate quality is maintained, thus it is recycled in the main stream process.

mainly because of their cost. New research should enable fast development of these materials for a wide range of uses (must, wine, concentrates).

This new research concerns the design of the inorganic membrane: chemical components of the hydrophilic membrane layer, adapting the pore size, decreasing the membrane thickness to limit the fouling with wine macromolecules and increasing flux with an efficient back-pulse system.

Electrodialysis for Ensuring Tartaric Stability of Wines

Electrodialysis (ED) is used to avoid the tartaric salts deposits at the bottom of bottles, that are not appreciated by the consumer. ED consists of extracting certain ions from wines, notably potassium, calcium and tartaric acid ions which contribute to reducing the level of over saturation of tartaric acid salts. The idea of stabilizing tartaric precipitation in wines through electrodialysis was proposed in the late 70s. ED has been re-examined as a truly efficient solution for facing the current constraints. Electrodialysis membranes for wine treatment have been designed and selected on the consideration of fouling resistance, selectivity and the extraction of Ca^{++} , K^+ , tartaric acid, economical and oenological requirements:

- Membranes must comply with regulations governing additives, materials in contact with foodstuffs and tests on the migration in hydro-alcoholic solution to an acidic pH;
- Kinetics of de-ionization adapted to potassium and calcium cations and tartaric anions; mechanical resistance; operational life of at least 2000 hours guaranteed for the anion exchange membranes which are usually the most fragile;
- No alteration of non-ionic compounds in wine and only slight modifications to the physico-chemical balance of the wine; maximum reduction of ethanol of 0.1% (v/v); pH reduction of less than 0.25 unit; reduction of volatile acidity of less than 0.009 g l^{-1} (expressed in sulphuric acid).

The assessment of tartaric stability of the wine relies on the analysis of the variation of conductivity, over time, of a sample at negative temperature and sprinkled with calibrated cream of tartar crystals while under constant agitation. This 4-hour test enables the evaluation of the drop in conductivity for a theoretically indefinite period. Then, the required conductivity reduction for the wine to become stable is achieved using an automatic controlled system. So that, after a maximum 4 h test it is possible to know the exact value of the conductivity of the wine to be treated to reach its equilibrium tartaric state. Then the electrodialysis system applied this value with an automatic and independent system.

This system is necessary for assuring a good quality. The knowledge of the composition of wine in K^{++} , Ca^{++} and tartaric acid does not allow the calculation of the amount of demineralization because of interactions between wine constituents and the effect of pH, alcohol and different organic acids. For example a wine with a high conductivity ($2500 \mu\text{s}$) may require a 15% conductivity removal, while a wine with a low conductivity ($1800 \mu\text{s}$) may require a 20%

conductivity removal. The wine is pumped into the tank and from here it circulates around the electrodialysis stack. When the conductivity reaches the critical value determined by the evaluation test, the volume of stabilized wine is fed into a reception tank. The next volume to be treated is then fed in and stabilized in the same conditions. This is a short discontinuous batch mode, the treatment being carried out over a few minutes on a small volume of wine. The treatment time and consequently the unit's performance directly depend on the wine's instability. The procedure is generally continuous and does not require fixed volumes of wine. The ion concentration in the concentrate circuit, which corresponds to the ionic extracts from the wine, is controlled by dilution using a threshold value in order to avoid cream of tartar crystals precipitating in the compartments within the membrane stacks.

As for other compounds contained in wine, detailed analyses have shown that polyphenols (anthocyanins and tannins), polysaccharides, amino acids and volatile compounds are not affected by the treatment. In comparative tests cooling modifies the state of the colouring matter more than electrodialysis. There were no significant sensorial differences between the control wine and the wine treated by electrodialysis⁹⁵⁻⁹⁶.

The reliability of electrodialysis of wines for preventing tartaric precipitation to occur, is based on a stability test integrated to an active control system, which determined for each wine both the treatment opportunity and the level of treatment. Compared to classical treatments, the electrodialysis option yields savings in filtering additives, seeding tartars or metatartaric acid, according to the technologies used. Waste from wine treatment is also reduced. The energy cost of tartaric stabilization by ED is especially low: total electricity consumption was between 0.5 and 1 kWh m^{-3} of treated wine, including pumping.

This is approximately ten times less than the energy required for classical techniques using refrigeration. Where the comparative analysis takes into account the fixed costs (five-year depreciation plus financing expenses) and the operating costs (energy consumption, consumable, membrane replacement, labour and maintenance), ED process is the winner. Costs per hectolitre treated can thus be reduced by 50 to 70% depending on the size of the installation, degree of automation (filling, emptying, rinsing, operational management) and the replacement frequency of membranes, compared to treatment at -3°C for 10 days.

Fourteen industrial treatment units of varying capacities (4000 to $10,000 \text{ l h}^{-1}$) have been operational in France, Italy and Spain since 1997 at wine merchant-maturers and producers. Sensory analysis over time and over a wide range of products confirmed the procedure's neutrality on organoleptic quality—it is extremely difficult to differentiate between wines before and after treatment. In Europe the electrodialysis system is authorized for table and country wines. Since 1995 much data has been collected on appellation wine in AOC—guaranteed vintage—area of wine production in France, Italy, Spain and Portugal. The excellent qualitative results on wine characteristics (stability and sensory analysis) allow this European AOC agreement to be planned from 1 August 2001. This regulation will be a good reference for other wines areas worldwide.

The innovative combination ED–MF process can solve the problems of microbiological stability, clarification and

Table 2. Comparison of traditional and membrane processes for wine making.

Traditional techniques for wine making for clarification and tartaric and microbiological stability		Novel procedure before bottling with membrane technologies	
Centrifugation	All of these operations are not used together. They are used separately according to the condition of the wine and the end result required.	Fining	
Fining		Electrodialysis	
Body feed		Cross-flow microfiltration	
Plate filtration			
Low temperature treatment			
Body feed or centrifugation with cyclones			
Pre-filtering membrane (dead-end)			
Sterilizing membrane			
Sequential process difficult to automate			24 h continuous process easily automated

tartaric stabilization with an excellent protection against oxidation in one step and in a continuous system, without any additive. In France in 2000, several wine producers used this ED+MF process for complying with expert quality criteria. It replaces all the stability and clarification treatments, both microbiological and tartaric, as shown in Table 2. This new treatment line is technically viable today and should be considered as the technological revolution of tomorrow, particularly when adopted by wine packaging centres. This technical innovation considerably simplifies the organization of wine treatment because it is a fully automated semi continuous process. It should, therefore, be systematically taken into consideration in the preliminary studies for new wine packaging units. It could also be efficiently integrated into existing installations where it would significantly increase the operational capacities of cold producing and 'sterilizing filtration' units at the end of the production line.

The requirements of an increasingly competitive export market with ever more stringent constraints mean that companies have to take steps to ensure optimal qualitative performance. In addition, problems related to environmental protection in wine production are also an incentive to adopt these new technologies. Technology is in fact the solution to the various problems that the winemaking sector faces.

MILK AND DAIRY INDUSTRY

Most of the industrial developments of membrane technologies in the food industry originate from the dairy industry. They have been more or less tightly linked to the progress in membrane operations: asymmetric organic membranes by the late sixties; composite inorganic membranes in the early eighties; porous ceramic membranes with multi-channel configuration in the early nineties which has enabled industrial application of the concept of uniform transmembrane pressure. UF is the most widely used process in the world dairy industry. The membrane area of RO has stabilized around 60,000 m², mainly for whey concentration. MF is developing due to its capability to retain, partly or totally, particles (micro-organisms, casein micelles, fat globules), and NF is given a large field of applications due to its intermediate selectivity (200–1000 Da) between UF and RO (demineralization, de-ionization, purification) in particular for whey protein valorization. The integration of membranes has been implemented throughout the milk and dairy processing chains—milk

reception, cheese making, whey protein concentration, fractionation of protein hydrolysates, waste stream purification and effluents recycling and treatment—all are membranes.

Control of Microbial Growth: Cross-Flow MF for Bacteria Removal From Milk (and Various Milk Products)

Like most foods, milk and its derivatives provide a favourable media for spoilage microorganisms. Consequently, pre-treatment as well as temperature–time parameters must be chosen in order to control microbial growth. Heat treatment and bactofugation are the most widely used processes for lowering the bacterial content of milk and milk products. They have advantages and drawbacks⁹⁷. MF for bacteria removal⁹⁸ takes advantage of both multi-channel ceramic membranes with highly permeable α -alumina support and the dynamic counter pressure concept⁹⁹, which maintains a low constant transmembrane pressure throughout the filtering path despite a high pressure drop created on the retentate side by a high cross-flow velocity (in the range 6–9 m s⁻¹). With a membrane of 1.4 μ m pore diameter the retention of bacteria is over 99.0% (decimal reduction 2.1–3.1 depending on bacteria morphology and volume), and casein transmission is around 99%. MF flux ranges from 500 to 700 l h⁻¹ m⁻² for 6–9 h.

The first industrial plant was installed in Sweden with the aim of increasing the shelf life of pasteurized milk¹⁰⁰, owing to the high retention of *B.Cereus* the shelf life became 16–21 days (from 6–8 days), and an improved flavour was noticed. Several hundreds of these systems (10,000 to 20,000 l h⁻¹) are currently running in Europe and North America for the manufacture of drinking milk. Due to high retention of *Listeria* and *Salmonella* as well as natural non pathogenic flora, the process is used for bacteria removal from milk prior to transformation into cheese (traditional AOP—guaranteed cheese—French cheeses from raw milk) or milk protein fractionation. Recent developments include:

- An alternative design of the process (2 stages) with final VRR = 200 (retentate fraction: 0.5%) which yields an increase in decimal reduction up to 3.6 and more;
- Little flux reduction due to a narrower pore size distribution of the 1.4 μ m membrane;
- A novel ceramic membrane with linear hydraulic resistance gradient¹⁰¹ which generates appropriate pressure

drop through the membrane without recirculating the permeate and ensures uniform constant flux of $500 \text{ l h}^{-1} \text{ m}^{-2}$ and decimal reduction of 4 for bacteria. Despite its higher manufacturing cost this novel membrane should lead to about 30% reduction of the investment cost required for bacteria removal by MF; a prospective improvement which could be generated by rotating dynamic¹⁰² or vibrating (oscillating flow regime) membrane filtration.

MF for Globular Milk Fat Fractionation

The separation of milk fat globule to two fractions (small globules $< 2 \mu\text{m}$ versus large globules $> 2 \mu\text{m}$) has been realized by a patented process using special microfiltration membranes^{103–104}. Large ranges of operating conditions have been tested: 1.8–10 μm pore size, 0.2–1.75 bar transmembrane pressure, 37–55°C temperature, 4–8 m s^{-1} tangential flow velocity and 2–20 volume reduction ratio. The raw material may be: whole milk, fat enriched milk, or cream. Transformation to drinking milks, yoghurts, sour cream, camembert, Swiss cheese and butters were also realized. It is claimed that, except butter, the use of the small globule fraction yields more unctuous products and finer textural characteristics compared to products made from untreated or large globule cream.

MF in Cheese Making

MF is used in an integrated protein extraction process for the manufacture of micellar casein products and whey proteins isolates. When skim milk is circulated along a MF membrane with a pore size diameter of 0.1 μm (ZrO₂, TiO₂ membrane on an Al₂O₃ support or C support) or of 0.2 μm (homogeneous Al₂O₃ membrane) a microfiltrate with a composition close to that of sweet whey is obtained. Moreover, it is crystal clear, sterile and free of phage particles that means it could be considered as virus-free, a property which could be useful for the medical uses of its derivatives. Concentration by ultrafiltration (UF membrane with a pore size $\leq 10,000$ Da) of this milk microfiltrate directly produces a whey protein concentrate (WPC) with a protein/total solid ratio of 77%, a value which can be increased to over 90% by diafiltration (whey protein isolate, WPI). Milk MF can be operated according to critical stability criterion (Flux/Shear stress) which allows the industry long runs with very moderate fouling when the proper operation mode (constant flux or constant transmembrane pressure with UTP system or static permeate counter pressure) is selected¹⁰⁵.

The retentate is an enriched solution of native and micellar calcium phosphocaseinate (NCPP)¹⁰⁶. It is purified by diafiltration against water and then vacuum evaporated and spray-dried^{107,108}. Native casein has excellent rennet-coagulating abilities. The coagulation time of a 3% NCPP solution is reduced by 53% compared to that of raw milk, and gel firmness at 30 minutes is increased by more than 50%¹⁰⁷. It shows many other interesting properties for the process cheese industry as well as for some odd applications: for example, it has a protective effect on equine spermatozooids.

Casein enrichment of cheese milk by membrane MF is expanding, especially in plants making hard cheese varieties, because it significantly improves rennet coagulability. Curds are firmer and consequently lead to fewer fines in whey, and induce a slight (1%) increased cheese yield. In this way, the economical productivity of plants is improved because of higher cheese production per vat and better added value of milk microfiltrate compared to that of classical whey.

In addition, the partial reduction of the ratio whey proteins/caseins, by MF significantly reduces the detrimental effects of heat treatment on rennet coagulability of milk.

Finally, native micellar casein and its co-product, the WPI obtained after the subsequent step of ultrafiltration of milk microfiltrate, are excellent starting substrates for further fractionation and isolation of individual caseins or whey proteins.

Nanofiltration, NF of Whey

Whey is a co-product of the cheese making and casein industry. The world production is close to 150 billion litres, which corresponds to more than 9 millions tons of dry matter. Whey composition depends on the production process, but roughly speaking, it may be considered as milk without casein and fat. Nutritional¹⁰⁹, biological¹¹⁰ and functional¹¹¹ properties of whey proteins make them attractive and explain why an actual whey industry has been developing over the last 25 years.

The preconcentration of whey on its production site is the major field of application of reverse osmosis (RO), in the dairy industry owing to its flexibility and low energy consumption (9 kWh t^{-1} water removed) as compared to vacuum evaporation (9 to 150 kWh). Whey is concentrated up to 18–27% dry matter. Beyond that range process performance is diminished due to the high osmotic pressure, high retentate viscosity, lactose crystallization and calcium phosphate precipitation. The high salt content of whey—8 to 20% Dry Matter—generates numerous processing difficulties: slowed lactose crystallization rate, fouling in MF and UF performed for manufacturing whey protein concentrates (WPC) and isolates (WPI) and spray-drying, nutritional imbalance in human and infant food. Thus, it is advantageous to demineralize whey before evaporation and drying.

Industrially, demineralization in the range 50% to 95% can be completed by electrodialysis (ED) or ion exchange (IE). Both processes have drawbacks: high investment and running costs, large volumes of polluting effluents (0.3–1.01 l^{-1} whey with biological oxygen demand, BOD₅ in the range 2.5–4.5 g l^{-1} in ED; 1.0–2.81 l^{-1} whey with 1.7 < BOD₅ < 2.2 g l^{-1} in IE). Nanofiltration makes it possible to achieve the concentration of dry matter (20–22% at VRR 4–5) and demineralization (25–50% and even 90% with a diafiltration step) in a single operation¹¹². The process is competitive to RO and ED. Moreover, the demineralization is more selective than with ED: reduction of monovalent (40–90%) and divalent nutritional value 5–20% for calcium and phosphates of ions as compared to 62% and 43% respectively for 50% demineralization in ED¹¹². Nonetheless, the selectivity of ED is altered by interactions between proteins and membranes under the effect of applied electric field, whereas at the highest

demineralization rate (88–90%), NF selectivity remains satisfactory (reduction in divalent ions 49–75% as compared to 80–90% with IE). Besides, losses of lactose and non-protein nitrogen are lower by 6% and 25% respectively¹¹³ than in ED¹¹⁴. Losses of proteins are smaller (1.7–8%) in NF¹¹⁵ than in IE (5–27% according to the type of whey and the demineralization rate achieved)¹¹⁶. These losses are due to protein adsorption on ion exchange resins, in particular a strongly acidic or alkaline one. In order to improve NF performance it is advised to limit fouling of the widely used spiral-wound modules by pretreating whey as for whey UF or MF and by selecting appropriate flux/shear stress ratio as in milk MF¹⁰⁵.

The introduction of an NF step significantly improves the technological characteristics of the concentrate and gives it higher value—increase in yield of lactose crystallization of 8–10% with a decrease by 2 to 3 (with a diafiltration step) of the hygroscopy of the powder obtained^{115,117}.

Extraction of Milk and Whey Proteins

During the past two decades, considerable interest has been paid to the development of sophisticated extraction procedures for milk proteins in order to satisfy the increasing needs of different industrial sectors for tailor-made products¹¹⁸. Due to the numerous potential applications of proteins in fabricated food products, increased attention is being directed towards large-scale extraction and purification of proteins and peptides from various sources.

Fractionation of the main whey protein first attempted in the mid-1980s when Pearce¹¹⁹ proposed exploiting the low heat stability of calcium-free α -lactalbumin, has recently undergone significant improvements. α -Lactalbumin is a calcium metalloprotein containing one mole of ionic calcium per mole of protein. Removal of calcium from α -lactalbumin by adjusting pH to about 3.8 or by the addition of a sequestering agent such as citric acid or sodium citrate¹²⁰ results in much reduced thermal stability compared to the native protein. Subsequent heating around 55°C for a limited period of time leads to a reversible and partially denatured form, which undergoes aggregation. This property of α -lactalbumin is used in two processes developed by Pearce¹²¹ and Maubois *et al.*¹²². The first one uses whey concentrated by ultrafiltration to about 12% total solids. pH is adjusted to 4.2 and heated at 65°C for 5 minutes to cause aggregation of α -lactalbumin; during this treatment, both immunoglobulins and the serum albumin also coprecipitate with α -lactalbumin. Separation of the precipitate is performed in a continuous desludging clarifier¹²¹. The supernatant is diafiltered through 50,000 Dalton cut-off ultrafiltration membrane to yield purified β -lactoglobulin. In the second process¹²² the thermocalcic aggregation process prior to UF concentration up to VRR 30, clarifies whey. A pH value of 3.8 and heating at 55°C for 30 minutes are used to allow co-precipitation of α -lactalbumin, immunoglobulins and bovine serum albumin. Separation of a highly purified (95%) soluble β -lactoglobulin is carried out by centrifugation or by MF (pore size 0.1 μm) with a diafiltration step. The sediment or MF retentate yields a 70%-purity α -lactalbumin fraction. Further improvement of the process and the purity of α -lactalbumin fraction are to be expected from a novel cascade of separations¹²³: pre-purification step by UF of defatted clarified whey¹²⁴; precipita-

tion step carried out on the ultrafiltrate. The purity of the final fractions was: 83–97% for α -lactalbumin and 0.98–0.96 for β -lactoglobulin. The co-products have higher value (WPI 99)¹²⁵ than those obtained with the process based on α -lactalbumin precipitation directly from whey¹²⁶.

α -Lactalbumin has a great potential market in nutritional foods because of its high content in Tryptophan (4 residues per mole, i.e., about 6%) and in infant milk formulas. Due to the fact that α -lactalbumin shows strong affinity for glycosylated receptors existing on the surface of ovocytes¹²⁷ and spermatozooids, it may also find use in therapeutic foods.

Since no biological function for β -lactoglobulin has been proposed yet, the only main uses of this protein appears to be in gel and foam-type products and in the manufacture of protein hydrolysates for food ingredients formulation.

Treatment of Dairy Effluents and Waste Streams

The effluents of waste streams stemming from milk and dairy processing represent a high potential of pollution: 0.2–111 effluents/l processed milk; polluting charge of 0.2–2.5 g l^{-1} biological oxygen demand (BOD) mainly due to loss of raw material (0.5 to 2% of milk processed)¹²⁸. Simultaneously, in spite of significant improvement over the last 10 years, water consumption still remains high: 0.2–111 l^{-1} milk. Municipal water cost steadily increases (1.2–5.7 € m^{-3} in France) and resources of appropriate quality tend to become scarce. Cleaning-in-place (CIP) operations contribute highly to water consumption and are responsible for 50 to 95% of the overall volume of waste streams and high pH (9–11) in waste water purification stations. Until now, the treatment has been operated in biological treatment stations. For a factory of a mean capacity (10^6 l milk/day), wastes are sludge and are usually used for landspreading (1 to 3 t dry matter) and water drained to rivers (0.3×10^6 to 3×10^6 l). Regular increase of taxes, severity of waste standard composition and EC regulations (by 2002 only ultimate wastes authorized in landfill sites, and by 2004 landspreading forbidden in sensitive zones) strongly weighs on the food and dairy industry. Industrial behaviour is changing from 'treat or pay' to 'treat or close'.

Over the past few years, the dairy industry has been attempting to find new processes using two different approaches:

- *Preventive approach*: This consists of separating and treating individual effluents at source. Several types of effluents are currently treated in industry: washing water of rennet casein precipitate (NF) and emmental cheese (dead-end filtration); white (flushing) and pre-rinsing waters (first step of CIP) (UF, NF, RO)^{129,130}. The outcome is a highly significant improvement of water quality after treatment (suspended solids, $\text{SS} < 2 \text{ mg l}^{-1}$; chemical oxygen demand, $\text{COD} < 35 \text{ mg l}^{-1}$; $\text{BOD}_5 < 3 \text{ mg l}^{-1}$) and the re-use of milk components dry matter (either as recycling back to the production unit or animal feed). Evaporation condensates ('cow's water') and milk and whey RO and NF permeates with COD content in the range 10 to 1000 mg l^{-1} , can be treated by using RO with eventual UF or MF pre-treatment (retention of SS and colloids)¹³¹. The RO permeate produced

(over 90% of the effluent treated, in the range 10^6 to 4×10^6 l/day) is used as water with 'food quality', for rinsing and cleaning operations and even as bottled water. Cheese brines are widely recycled after a MF step, which strongly reduces microbiological counts, without altering the chemical composition (nitrogen, ash) contrarily to conventional pasteurization¹³². Finally, alkaline and acid CIP solutions are periodically drained to waste due to their pollution load (SS, COD, etc.). The periodicity depends on the CIP processing type: single use re-use or multi-use. For a factory which transforms 10^6 l milk/day, the total amount of caustic soda drained to waste represents around 120 year^{-1} (recall: 125×10^9 l milk processed in the EC). Using MF and UF^{133–134} or NF¹³⁵ cleaning solution can be regenerated. Performances of NF are promising with regard to pollution reduction (SS: 100%; $-20\% < \text{COD} < 60\%$), $500 \text{ l h}^{-1} \text{ m}^{-2} < J < 70 \text{ l h}^{-1} \text{ m}^{-2}$ and caustic soda recovery (98–99%). The retentate, which contains highly concentrated nitrogen and fatty compound is treated as a sludge which should be given added value. More than 30 plants are running worldwide: 20 MF^{136,137}; 5 UF¹³⁸; 8 NF^{139,140}. The pay-back claimed by the manufacturers is in the range $< 1\text{--}5$ years, which is far shorter than the expectations assessed from research results — 7.7 years¹³³ or more¹³⁵. Nevertheless, the likely evolution of waste discharge regulations (Na, etc.) should soon make an integrated membrane regeneration process compulsory.

- *Curative approach:* Several companies propose the use of a membrane aerobic bioreactor (MBR) for the treatment of end-of-pipe municipal and industrial wastewaters. Two designs of MBR exist: external loop with organic or ceramic UF membranes; hollow fiber UF membranes immersed in the station. Membrane separation replaces the decantation practised in classical waste treatment and allows the capacity of the latter to be increased requiring small extra 'floor' area. The manufacturers claim a significant reduction of sludge ($\times 0.3\text{--}0.5$ with a conversion rate in the range $0.1\text{--}0.4 \text{ kg dry sludge kg}^{-1} \text{ COD removed}$) and enhanced quality and stability of cleaned-up water: sanitized, $6.5 < \text{pH} < 7.5$, $\text{SS} = 0$, $\text{COD} < 60 \text{ mg l}^{-1}$ total Kjeldhal nitrogen $< 10 \text{ mg l}^{-1}$. The investment costs are heavy, 1 Million € for an external organic plate-and-frame MBR ($300 \text{ m}^{-3} \text{ d}$) (2.5 M € for an external ceramic multichannel MBR ($600 \text{ m}^{-3} \text{ d}$). With regards to energy consumption, the reference for common wastewater purification station is $1.5\text{--}7.0 \text{ kWh m}^{-3}$ as compared to $0.1\text{--}0.2 \text{ kWh m}^{-3}$ with immersed membranes and $2\text{--}10 \text{ kWh m}^{-3}$ with external membranes. Very few membrane bioreactors are currently running for the purification of waste streams in the food industry, but researches are running these aerobic MBRs as well as anaerobic MBRs (which theoretically should yield much lower sludge rates with the objectives of achieving high active biomass concentration, minimum apparent growth of micro-organisms, adapting micro-organisms to the specific composition of food waste streams (variable with the industrial field) and optimizing the coupling fermentation—membrane separation (minimizing fouling, filtration stability at high cell density conditions).

In summary, membrane processes provide the dairy industry with reliable safe, clean and sober processes. The dairy industry has now got the principles for extracting proteins from milk and whey, single or in a mixture, with more or less high purity, which fit well the needs in food and non-food applications. UF and MF are particularly valuable for improving traditional cheese making and creating new types of cheeses, which answer well the continuous need of novelty of most of the consumers. Extraction procedures developed for the separation and purification of milk components have interesting functional or biological properties. Among the available operations, membrane separations appear to be the most suitable because they are modular and can operate at moderate temperatures.

Milk is unquestionably a unique source of high-quality protein imparting both nutritional and functional characteristics to foods. Due to the amount of research that has been focused on the improvement of separation processes, it is likely that the general quality and production efficiency of the various milk protein ingredients, i.e., total milk proteins, acid and rennet casein, and whey protein concentrates, will increase in the near future. The development of cross-flow microfiltration will also facilitate commercialization of native micellar casein and its derived co-product, a unique whey protein isolate. Both products in turn can be used as new raw materials for the separation and purification of individual caseins and whey proteins.

CONCLUSION

In the whole food industry, membrane processes have contributed to the revision of traditional flow sheets in the processing of fluids with the major issues of simplification, improvement of competitiveness, process or product novelty and environmental friendliness. Recent industrial applications have been developed for fruit, vegetable and sugar juices, beverages (based on vegetable proteins, beer, and wine) and milk and dairy products. Further integration of membrane operations is to be expected, provided they are designed in such a way that at each processing step, end products, co-products and wastes are given even attention. The safety and quality of the products manufactured must be ensured with regard to microbiological, functionality, texture, flavour and taste aspects. To achieve this, the physico-chemical, rheological and structural characteristics of food components are to be well studied. In consideration of this, the food industry will have excellent means in creating high added value by manufacturing novel foods or beverage with health value (nutritional, biological, etc.) specifically addressed to different kinds of consumers—infants, children, the elderly, young, sportsmen. Simultaneously, membrane processes will provide the food industry with efficient tools for limiting its environmental impact. There is no doubt that in close future any food process will include at least one membrane operation, as is already the case in drinking water production.

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