3
Milk and Dairy Effluents Processing: Comparison of Cross-Flow and Dynamic Filtrations

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3.1
Introduction

The dairy industry is very important in Europe where it represents 14% of agricultural national production [1]. The European dairy industry is famous for the quality of its products, especially for its variety of cheeses and yogurts, dairy cream, ice creams, and so on. Milk is a complex fluid and an important source of proteins. The average composition of milk is given in Table 3.1 [2]. As noted by Brans *et al.* [3], the functionality of milk proteins is larger if they have been separated and purified. Thus, their fractionment leads to more efficient and diversified applications.

3.1.1
Properties and Applications of Various Proteins

3.1.1.1 Caseins

This protein (24 kDa in molecular size) is generally aggregated as micelles, which average 110 nm in size (or about 300 kDa). Several casein species exist, α_1, α_2, β, $\varkappa$. Concentrated casein solutions can be mixed with cream for production of cheese and for standardization of milk composition, required for industrial cheese production (between 36 and 45 g/L). They are also used for infant formula and as emulsifiers. Dried native caseins can also serve as food additive [4, 5]. Casein β and $\varkappa$ can be separated from sodium caseinate.

3.1.1.2 Whey Proteins

The main proteins are α-Lactalbumin (α-La, 14 kDa) and β-Lactoglobulin (β-Lg, 36 kDa in dimer form), which represent 70% of total whey proteins. α-La has several pharmaceutical applications and is added to infant milk while β-Lg can be used for emulsification, foaming and gelling [6, 7] and can replace egg albumin in food products. It is also used as an additive in energetic drinks or in meat and fish based-products. Bovine serum albumin (BSA, 66 kDa) can be used for foaming and gelling in human food [8]. Lactoferrin (86 kDa) is used in cosmetics for skin protection and as anti-bacterial in meat preservative and in parenteral feeding [4, 7].

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Table 3.1 Average composition of cow milk: concentration and size distribution.

	Concentration in whole milk (g/L)	Size range and average (at weight average)
Water	87.1	
Fat globules	4.0	0.1–0.15 μm, average 3.4 μm
Casein (in micelles)	2.6	20–300 nm, average 110 nm
Serum proteins	0.7	3–6 nm
α-Lactalbumin	0.12	14 kDa
β-Lactoglobulin	0.32	18 kDa
BSA	0.04	66 kDa
Proteose-peptone	0.08	4–40 kDa
Immunoglobulins	0.08	150–900 kDa
Lactoferrin	0.01	86 kDa
Transferrin	0.01	76 kDa
Others	0.04	
Lactose	4.6	0.35 kDa
Mineral substances	0.7	
Organic acids	0.17	
Other	0.15	

3.2 Applications of Membrane Cross-Flow Filtration to Milk Processing

3.2.1 Milk Microfiltration

The main applications of MF to milk include bacteria and spore removal (cold pasteurization) and production of casein concentrates for milk standardization or cheese production with addition of cream. Milk is filtered after its fat has been removed in order to avoid unnecessary membrane fouling.

3.2.1.1 Bacteria and Spore Removal

This process does not heat denaturate whey proteins and provides longer preservation than pasteurization. However, it is necessary to transmit through the membrane all proteins, which is difficult, due to the large micelle size and internal membrane fouling. A commercial process, Bactocatch, has been proposed by Alfa Laval (France), which consists [9] in combining large milk velocities (6–8 m s^{-1}) with a low uniform transmembrane pressure (TMP) in a ceramic tubular membrane with 1.4 μm pores. The uniform TMP is obtained by a cocurrent permeate recirculation with a pump to produce the same pressure gradient on both sides of the membrane and this process is known as UTP (or UTMP) mode [3]. Later, Isoflux tubular ceramic membranes with a continuous reduction in membrane thickness to reduce filtration resistance along the membrane at the same rate as TMP have been proposed by TAMI Co (Nyons, France) [3]. SCT (now Exekia, Bazet, France) introduced Membralox ceramic

membranes with a porosity gradient (GP) to achieve uniform flux [3]. These two types of membranes do not require permeate recirculation and are therefore more economical in energy. Saboya and Maubois [10] reported a decimal log bacterial reduction of more than 3.5 with the Bactocatch system.

3.2.1.2 Casein Micelles Separation from Whey Proteins

Unlike the case of bacterial removal, casein should be rejected by the membrane and pore sizes are smaller, from 0.2 to 0.05 μm, but the same type of systems with uniform TMP (UTP) or uniform flux along the membrane at high fluid velocity have been used for this application.

Daufin *et al.* [11] have used SCT membranes with 0.1-μm pores in the UTP mode with cocurrent permeate recirculation to separate caseins from whey proteins and obtained a whey-protein transmission of 70–80%. Gésan-Guiziou *et al.* [12], using a similar ceramic membrane (Kerasep 0.1 μm, TechSep Miribel, France) and the same filtration bench in UTP mode reported fluxes at 50 °C of about 80 $L\,h^{-1}\,m^{-2}$ with 50–80% α-La transmission, but permeate turbidity was relatively high (100–200 NTU), corresponding to about 2% casein transmission. Pouliot *et al.* [13] obtained permeate fluxes of 90 $L\,h^{-1}\,m^{-2}$ at fluid velocity of 6.9 $m\,s^{-1}$ and a TMP of 190 kPa with a 0.22 μm pores Ceraflo ceramic membrane at a volume-reduction ratio (VRR) of about 1.5. Vadi and Rivzi [14] compared UTP and non UTP modes with a 0.2-μm pore ceramic Membralox multichannel membrane (Exekia, France). They obtained, in UTP mode, a flux of 70 $L\,h^{-1}\,m^{-2}$ at a VRR of 4, a TMP of 193 kPa, and a fluid velocity of 7.2 $m\,s^{-1}$. They found that the non-UTP mode gave higher flux up to a VRR of 4, while the UTP mode performed better at higher VRR. They also observed that the cake formed during MF in non-UTP mode was more difficult to erode than the cake produced under UTP conditions. Le Berre and Daufin [15] obtained a 99.5% casein retention at a flux of 100 $L\,h^{-1}\,m^{-2}$ with a 0.1-μm pore ceramic membrane and a whey-protein transmission between 70 and 90%. Samuelson *et al.* [16] used a 0.14-μm pore ceramic tubular membrane (Orelis, France) for casein concentration from skim milk, while minimizing whey-protein rejection by using cross-flow velocities up to 8 $m\,s^{-1}$. They reported a maximum flux of 145 $L\,h^{-1}\,m^{-2}$ at a speed of 8 $m\,s^{-1}$ and 55 °C, which fell to 80 $L\,h^{-1}\,m^{-2}$ at 4 $m\,s^{-1}$. Whey-protein transmission was 88%, at 8 $m\,s^{-1}$ and 74% at 6 $m\,s^{-1}$, but casein rejection was low at 90%. A recent investigation of casein concentration by MF using polymeric membranes was made by Lawrence *et al.* [17] who used 0.3- and 0.5-μm pore PVDF (polyvinyliden fluoride) membranes, both in a flat-sheet laboratory module and in a spiral wound industrial pilot in non-UTP mode. They observed a casein rejection that increased from 96% at a TMP of 50 kPa to 98% at 150 kPa and 100% at 258 kPa. β-Lg transmission decreased from 22% at 50 kPa to 8% at 150 kPa and 1% at 258 kPa. In the flat-sheet module at 50 °C and a velocity of 0.44 $m\,s^{-1}$, the permeate flux decayed from 60 $L\,h^{-1}\,m^{-2}$ to 52 $L\,h^{-1}\,m^{-2}$ over a period of 2 h. In the spiral module at the same velocity and 40 °C, the flux remained steady with time, at near 32 $L\,h^{-1}\,m^{-2}$.

Nelson *et al.* [18] developed a multistage MF process to remove a high percentage of whey proteins from skim milk while producing a low concentration factor retentate

from microfiltration. The microfiltration retentate was blended with cream to standardize milk for traditional Cheddar cheese making. The MF permeate was ultrafiltrated and the permeate obtained from this ultrafiltration was diafiltered in order to remove whey proteins from skim milk before cheese making. The total process had 3 stages: the first consisting in a MF of skim milk up to a VRR of 3, the second one was a first diafiltration (DF) of permeate from ultrafiltration and the last one was a second diafiltration. They used a UTP pilot (Tetra Alcross M7, Tetra Pack, Denmark) equipped with 0.1-μm pore ceramic membranes (Membralox). The TMP was maintained between 23–28 kPa. MF flux was 30 $L\,h^{-1}\,m^{-2}$. They removed about 95% of whey proteins.

Zulewska *et al.* [19] microfiltered pasteurized skim milk using several systems. The first was a UTP pilot-scale with a ceramic 0.1 μm (Membralox, Pall Corp., East Hills, NY). The second was a 0.1-μm alumina membrane with graded porosity (GP, Membralox, Pall Corp.), and the third a polyvinylidene fluoride (PVDF) spiral-wound (SW) module with 0.3-μm pores (Parker-Hannifin, Tell City, Ind., USA) membranes. They found differences in flux among ceramic UTP, ceramic GP, and polymeric SW microfiltration membranes (54.08, 71.79, and 16.21 $kg\,m^{-2}$ per hour, respectively) when processing skim milk at 50 °C in concentration tests until a concentration factor of 3 was obtained. These differences in flux among the membranes would influence the amount of membrane surface area required to process a given volume of milk in a given time. The protein contents of microfiltration permeates from UTP and GP membranes were higher than from SW membranes (0.57, 0.56, and 0.38%, respectively). Casein transmission in permeate was highest for the GP membrane and minimum in UTP module. The efficiency of removal of serum proteins was 64.40% in UTP mode, 61.0% and 38.6% respectively for GP and SW membranes. The SW polymeric membranes had a much higher rejection of serum proteins than the ceramic membranes.

These data will be later compared with those obtained using dynamic microfiltration.

3.2.2
Milk Ultrafiltration (UF)

Ultrafiltration is used extensively in the dairy industry for concentrating proteins in cheese production by membrane [20, 21] and for the recovery of soluble proteins from whey [22]. A recently emerging application is the fractionation of whey proteins, mostly α-La and β-Lg [23, 24] for increasing their concentration in cheese or as food additives. This fractionation was previously achieved by chromatography, which gave a high purity, but a low output.

3.2.2.1 Total Proteins Concentration

In order to retain, at least partially, α-La, the smallest whey protein, membranes must have a cut-off between 5 and 20 kDa. Clarke and Heath [24] have ultrafiltered skim milk using 5 kDa polysulfone spiral-wound modules. Their permeate flux was 14 $L\,h^{-1}\,m^{-2}$ at 225 kPa and a cross-flow velocity of 0.3 $m\,s^{-1}$. Labbe *et al.* [22]

recovered and concentrated soluble proteins from whey by UF with a 20-kDa Carbosep membrane (zirconium oxide on carbon support, Techsep, Miribel, France). Permeate fluxes were higher than for skim milk, but decayed during the first hour of filtration, due to protein–ZrO_2 interactions.

Yan *et al*. [25] ultrafiltrated whole milk using tubular membranes (HBJ 180, Abcor Inc, USA). They obtained a maximum flux of 42 $L\,h^{-1}\,m^{-2}$ at 100 kPa, 49 °C and a fluid velocity of 3.13 $m\,s^{-1}$. The permeate flux decayed linearly with VRR from 29 $L\,h^{-1}\,m^{-2}$ at VRR = 1 to 13 $L\,h^{-1}\,m^{-2}$ at a VRR of 2.8.

3.2.2.2 **Whey-Protein Fractionation**

Due to the difficulty of separating proteins with similar size such as α-La and β-Lg, most tests were not done on milk, but on binary protein mixtures or on protein concentrates. Cheang and Zydney [26] studied the separation of α-La and β-Lg from a binary mixture of these two pure proteins in a NaCl solution prefiltered at 0.2 μm, using diafiltration (DF). This DF was performed with a small Amicon stirred-cell equipped with a 30-kDa cellulose membrane, at two pH of 5.5 and 7.2. With the 30-kDa membrane, α-La transmission was 26% at a permeate flux of 12 $L\,h^{-1}\,m^{-2}$ against only 0.5% for β-Lg. These transmissions increased with increasing ionic strength to reach 60% for α-La at a strength of 150 mM at pH = 5.5, and 40% at pH = 7.2. β-Lg transmissions were maximum at pH = 7.2. Selectivity (ratio of α-La to β-Lg transmissions) reached a maximum of 58 at a pH of 5.5 and an ionic strength of 50 mM, but it decreased to 35 when permeate flux was doubled. With a 50-kDa PES (Polyether sulfone) membrane, the maximum selectivity dropped to 10.5 at pH = 5.5 and an ionic strength of 150 mM, due to the larger zeta potential of this membrane. The authors concluded that it was possible to separate α-La and β-Lg proteins with a high selectivity and a high yield rate, by optimal choices of pH, ionic strength and membrane cut-off. In a subsequent paper [27], the same authors obtained purified α-La and β-Lg fractions from whey protein isolate with a two-stage process. The first step was a diafiltration at 100 kDa to separate α-La and β-Lg in permeate from BSA in retentate. The second step was an ultrafiltration of permeate at 30 kDa followed by a DF in order to separate β-Lg in retentate from α-La in permeate. After 10 diavolumes, 75% of α-La was recovered in permeate. The final selectivity was 21 at the end of second DF. They compared this process with a second one in which the first DF was made at 30 kDa to collect α-La in permeate, while retentate was diafiltered at 100 kDa to collect β-Lg in permeate. This second process gave a higher α-La concentration than for the first process, but a smaller yield, 85% instead of 95%.

To produce purified α-La from acid casein whey, Muller *et al*. [28] proposed a prepurification step by UF with a limited transmission of β-Lg. Membranes tested were a 150-kDa Carbosep M1 and ceramic ones (TAMI) of 150, 200 and 300 kDa. With the M1 membrane, α-La transmission decayed from 80% at 0.5 bar and a flux of 30 $L\,h^{-1}\,m^{-2}$ to 58% at 3 bar when permeate flux rose to 80 $L\,h^{-1}\,m^{-2}$. Transmissions were lower for the 300-kDa TAMI membrane and decayed with VRR from 35% at VRR = 1.5 to 25% at VRR = 4. They obtained a α-La yield in permeate of 53% and a purity (ratio of individual to total protein concentration) of 0.44 for a VRR of 9 with a

permeate flux of $30\,L\,h^{-1}\,m^{-2}$. β-Lg transmission was 6% at a VRR of 3.5, and dropped to 4% at VRR = 8. Their conclusion was that variations of physicochemical and hydrodynamic conditions could induce large differences in protein transmission.

Almécija *et al.* [29] investigated the effect of pH (from 3 to 10) on the fractionation of whey proteins by diafiltration using a 300-kDa tubular ceramic membrane. α-La and β-Lg were collected in permeate while the retentate was enriched in BSA, immunoglobulins (Ig) and lactoferrins. Lowest permeate fluxes were obtained at pH 4 and 5, the isoelectric point of α-La and β-Lg, due to increased fouling by aggregates of uncharged protein molecules, while the highest were obtained at pH 9 and 10, since membrane protein repulsion decreases aggregation and fouling. The largest yields of α-La in permeate (58%) were obtained at pH 7–9, and the lowest (4%) at pH 4. For β-Lg, the permeate yields followed the same trend, but were lower, 33% at pH 8 and 9 and 2% at pH of 4 and 5.

Bramaud *et al.* [30] presented a process based on selective precipitation of α-La by heat treatment at 55 °C for 30 min at pH of 3.9 followed by a centrifugation for separating in the soluble phase lactose and β-Lg from a precipitate containing BSA, Ig and α-La. In the second step, lactose was separated from β-Lg by diafiltration, at 0.5 μm while the precipitate was resolubilized with addition of $CaCl_2$ to obtain a final yield of 57% for α-La. Lucas *et al.* [31] obtained a maximum transmission of 37% for α-La and 10% of β-Lg, corresponding to a selectivity of about 3 using a 50-kDa Carbosep membrane.

3.2.3
Applications of Milk Nanofiltration (NF) and Reverse Osmosis (RO)

3.2.3.1 Treatment of Cheese Whey and Fabrication of Yogurts

Cheese whey is generated by the traditional cheese fabrication consisting in coagulation of cream and casein. Each kilogram of cheese produces 5–10 kg of whey that contains about $6\,g\,L^{-1}$ of serum proteins, $48\,g\,L^{-1}$ of lactose and $6-13\,g\,L^{-1}$ of minerals. It is preferable to treat it as it constitutes a high COD (Chemical Oxygen Demand) effluent and the proteins and lactose it contains can be recovered in the food and animal feed industry, after demineralization by electrodialysis or ion exchange. In order to save transportation costs, whey can be concentrated by RO or by evaporation before a two-stage treatment using UF to concentrate proteins in the first retentate followed by NF to recover lactose in second retentate. Alternatively, a single NF step permits to concentrate serum proteins to 22% at VRR = 4.5, while reducing the amount of minerals by 25–50% [32]. These serum proteins can be spray-dried and used in various food applications under the names of whey protein concentrate (when containing 35–80% of proteins) or whey proteins isolates (with 80–95% of proteins) [3].

Nanofiltration has been used as an alternative to vacuum evaporation for concentrating milk in fabrication of yogurts, as it requires less energy. It is also used for selective demineralization of yogurts, for instance to lower sodium concentration or enrich them in magnesium or iron [20]. It is then possible to make low-fat yogurts

with better organoleptic properties than classical ones. But the main application of NF and RO seems to be the treatment of dairy process waters and effluents, in order to recover milk proteins and lactose, while obtaining a depolluted permeate that can be recycled as water for rinsing or cooling if its ionic and lactose content has been sufficiently lowered.

3.2.3.2 Treatment of Dairy Effluents

Dairy industry process waters resulting from starting, stopping or rinsing phases in the cheese-making process constitute a major source of milk protein loss as well as of pollution [33]. The chemical oxygen demand (COD) content of these effluents, mainly due to the presence of lactose [34], is high, ranging generally from 500 to 6000 $mgO_2\,L^{-1}$. Most of the earlier work on this process has been done using NF or RO spiral-wound modules [34–36] because of their availability and relatively low cost. Balannec *et al.* [34], using milk diluted three times with an initial COD of 36 000 $mgO_2\,L^{-1}$ as an effluent model with a spiral-wound module equipped with an Osmonics Desal 5 DL membrane of 150–300 Da cut-off. They obtained permeate fluxes ranging from 24 $L\,h^{-1}\,m^{-2}$ at initial concentration, a temperature of 25 °C and a transmembrane pressure of 1900 kPa to 12 $L\,h^{-1}\,m^{-2}$ at a volume-reduction ratio (VRR) of 5. The corresponding permeate COD rose from 125 $mgO_2\,L^{-1}$ at VRR = 1 to 400 at VRR = 5, remaining above the allowed French rejection limit of 125 $mgO_2\,L^{-1}$. Better COD removal was achieved when these authors used a Koch TFC HR reverse osmosis membrane that yielded a permeate COD of only 60 $mgO_2\,L^{-1}$ at VRR = 5, but the corresponding permeate flux fell from 18 $L\,h^{-1}\,m^{-2}$ at VRR = 1, to 7 at VRR = 5. These permeate fluxes were low because spiral-wound modules have a small hydraulic diameter (0.5 mm), and the high viscosity of concentrated milk prevented reaching high VRR. Vourch *et al.* [36] treated selected waste waters collected form dairy plants with a RO Koch TCR spiral-wound module in order to obtain recyclable water. Their permeate flux decayed from 30 $L\,h^{-1}\,m^{-2}$ at VRR = 1 to 9 at VRR = 5. They concluded that a RO + RO cascade permitted to obtain a recovery 90–95% of water recyclable as boiler feed with a highly charged effluent, against a single RO step for a low charged one. The total organic carbon in purified water was lower than 7 $mg\,L^{-1}$, against an initial value of 1000, while the conductivity was $< 50\,\mu S\,cm^{-1}$.

3.3 Dynamic Filtration

3.3.1 Principle and Advantages of Dynamic (Shear-Enhanced) Filtration

We have seen in previous sections that in milk MF it was important to increase membrane shear rate by using high fluid velocities while keeping TMP low and uniform, in order to transmit proteins through the membrane. This could only be achieved with permeate recirculation or specially designed membranes and the

energy necessary to drive recirculation pumps was high. In whey protein fractionation by UF, the TMP had to be limited to retain sufficient transmissions and permeate fluxes were often low, from 25 to 30 $L\,h^{-1}\,m^{-2}$. A RO stage was necessary to achieve sufficient COD reduction in treatment of dairy process waters, leading to low flux and high cost.

Dynamic or shear-enhanced filtration consists in creating the shear rate at the membrane by a disk rotating near a fixed circular membrane or by rotating circular membranes around its axis or by vibrating the membrane either longitudinally or torsionally around a perpendicular axis [37]. This mode of filtration can generate very high shear rates at the membrane that not only increase substantially the permeate flux, but have a favorable effect on membrane selectivity. Microsolute transmission is increased in dynamic microfiltration, which reduces cake formation by combining a high shear rate with a low TMP. In addition, high shear rates reduce concentration polarization and the concentration of rejected solutes at the membrane. Thus, concentration gradient and diffusive solute transfer through the membrane are decreased, which increases solute rejection rates in NF and RO, when mass transfer through the membrane is mainly diffusive. At the same time, permeate fluxes keep increasing until high pressures, as the pressure-limited regime is extended by the reduction of concentration polarization and very high fluxes can be obtained at high TMP. The inlet flow rate into the module needs to be only slightly larger than the filtration flow rate, reducing pumping energy.

The drawbacks of dynamic filtration are its complexity and limited membrane area for some systems, such as multicompartment rotating-disk systems, which raise the equipment cost. But, the recent availability of large-diameter ceramic disk membranes permits the construction of immersed rotating membranes of 80 m^2 area or more in a single housing, which are easier and less costly to build than multicompartment systems.

3.3.2
Industrial Dynamic Filtration Systems

The first commercialized dynamic filtration systems were of Couette flow type with cylindrical membranes rotating inside a concentric cylindrical housing, such as the Biodruckfilter (Sulzer AG, Winterthur, Switzerland) and the Benchmark Rotary Biofiltration (Membrex, Garfield, NJ, USA) [38]. This concept takes advantage of Taylor vortices created at large speed in the annular space between membranes and housing that increase the shear rate, but the maximum membrane area of commercial systems is about 2 m^2.

The Dyno system, manufactured by Bokela GmbH (Karlsruhe, Germany), consists in several disks rotating on the same shaft between fixed circular membranes for a total membrane area up to 8 m^2. Its maximum pressure is 600 kPa (Figure 3.1). It is available with polymeric or ceramic (metallic) membranes. The Optifilter CR (Metso Paper Raisio, Finland) features blades rotating between stationary flat circular membranes with a tip azimuthal speed of 10 to 15 $m\,s^{-1}$. Its total membrane area can exceed 140 m^2 with a 132-kW motor [39]. They are used

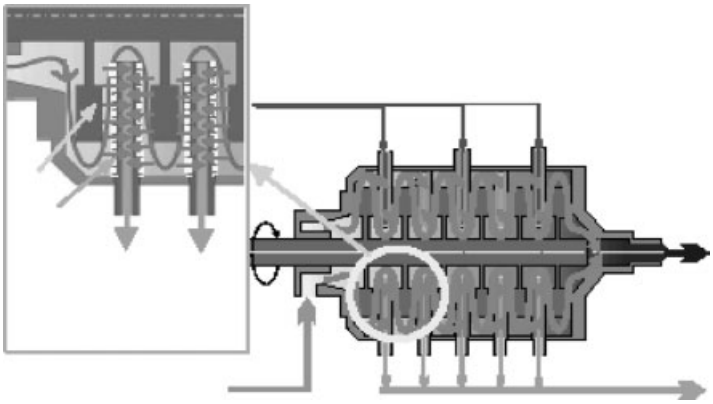

Figure 3.1 Dyno rotating-disk module (Bokela, Germany).

by more than 30 plants, mostly for treatment of pulp and paper effluents or pigment recovery.

The recent availability of ceramic membrane disks, especially in Germany, has spurred the commercialization of multishaft systems with overlapping rotating membranes. For instance, the MSD (Multi Shaft Disk) system (Westfalia Separator, Aalen, Germany) features 31.2 cm diameter ceramic membranes mounted on 8 parallel shafts arranged as shown in Figure 3.2. All disks rotate at the same speed and are enclosed in a cylindrical housing. Other systems, the Rotostream (Canzler, Dueren, Germany) [40] and the Hitachi (Tokyo, Japan) available up to, respectively, 150 and 100 m^2 membrane area have their parallel axes in the same plane. The Novoflow Company, (Oberndorf, Germany) manufactures single-shaft rotating MF and UF ceramic membranes systems, the SSDF (Single Shaft Disk) using 312-mm ceramic disks for a membrane area of 15 m^2 per module. The company reported a low energy consumption of 2.5 kW for a 15-m^2 module, corresponding to 0.64 € per m^3 of permeate and a total operating cost of 7.4 €/m^3. The SSDF is also available with composite MF-UF-NF membranes of 55 cm diameter with 25 m^2 of membrane per module.

Krauss-Maffei Process Technology (KMPT AG, Germany, www.kmpt.com), has developed a dynamic filtration module, similar to the MSD, but which can be equipped with rotating ceramic or polymer membrane disks. The module is in stainless steel and has a membrane area of up to 16.4 m^2. Membrane pore sizes range from 7 nm to 2 μm.

A vibratory membrane system (VSEP, New Logic Emeryville, Ca, USA), consists of a stack of circular organic membranes (Figure 3.3), mounted on a vertical torsion shaft spun in azimuthal oscillations by a vibrating base, at its resonant frequency of about 60 Hz. The shear rate at the membrane is produced by the inertia of the retentate that moves at 180° out of phase with the membrane and varies sinusoidally with time. The use of resonance minimizes the power necessary to produce the vibrations, which is only 9 kW, even for large units of 150 m^2 membrane area (Figure 3.4) The key parameter governing performance is the maximum azimuthal displacement of the

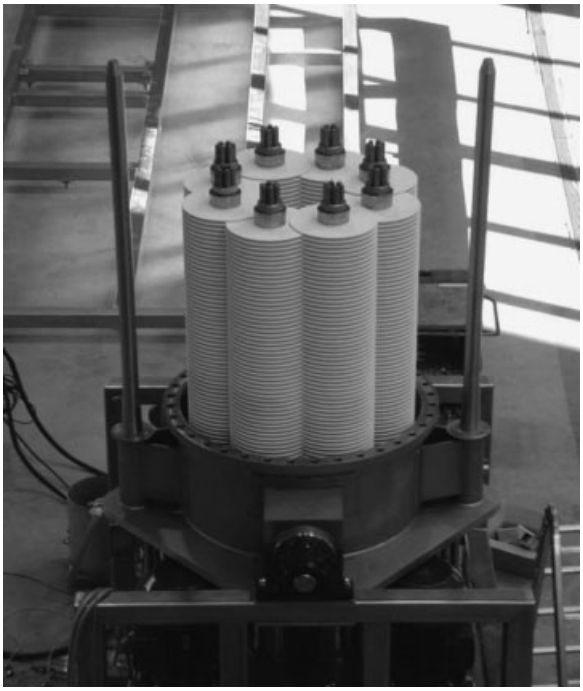

Figure 3.2 Industrial MSD module with 8 parallel shafts and 31-cm ceramic disks. Courtesy of Westfalia Separator.

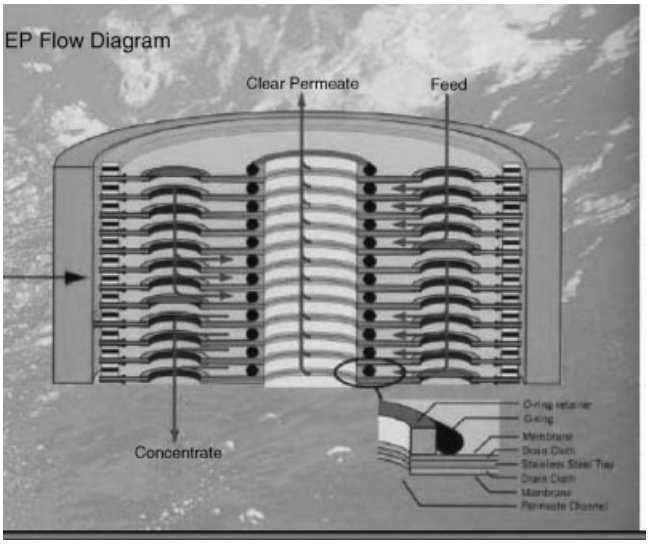

Figure 3.3 Schematic of circulation in VSEP membrane stack (Courtesy of New Logic Research).

Figure 3.4 Industrial VSEP vibrating modules (Courtesy of New Logic Research).

membrane rim, which has been measured as a function of frequency in [41] and is limited to about 3 cm. The VSEP has been used for the first time in Europe in 2007 to treat anaerobically digested pig manure. The system was installed and commissioned in Belgium at a major pig farm where it will be used for the biomethanation of raw manure, a comprehensive process developed by the Belgian firm where methane is recovered and converted into electrical energy. Zouboulis and Petala [42] studied the performance of VSEP for the treatment of raw stabilized leachate produced during landfill of municipal wastes. Four different membrane types were examined for the treatment of leachates, that is, one for microfiltration (0.1 μm), two for ultrafiltration (100 and 10 kDa) and one for nanofiltration (50% rejection of NaCl). The removal of organic matter in terms of COD value exceeded 60% for all cases.

The PallSep (Pall Corp, USA) is Pall's version of the VSEP intended for biotechnological and food applications and is available with up to 32 m^2 of membrane area. Postlethwaite *et al.* [43] investigated this system for protein recovery from a model biological feed stream containing 200–500 g L^{-1} *Saccharomyces cerevisiae* and 0.75 g L^{-1} bovine serum albumin (BSA). They reported that the flux and transmissions at a biomass concentration of 500 g L^{-1}, were 45 L h^{-1} m^{-2} and 67%, respectively, and could be maintained over extended periods.

3.3.3
Application of Dynamic Filtration to Skim-Milk Processing

3.3.3.1 Casein Separation from Whey Proteins by MF

One of the first applications of dynamic filtration to this task in UHT milk has been made with a VSEP pilot [44] equipped with a 500-cm^2, 0.1-μm pore Teflon membrane using UHT milk. The permeate flux at 45 °C and maximum vibration frequency (60.75 Hz) reached a plateau of 95 L h^{-1} m^{-2} at 100 kPa. This flux decayed with time to 50 L h^{-1} m^{-2}, which corresponded to the critical flux for stable operation. Permeate turbidity decayed with time from 52 to 15 NTU, indicating very good casein micelle

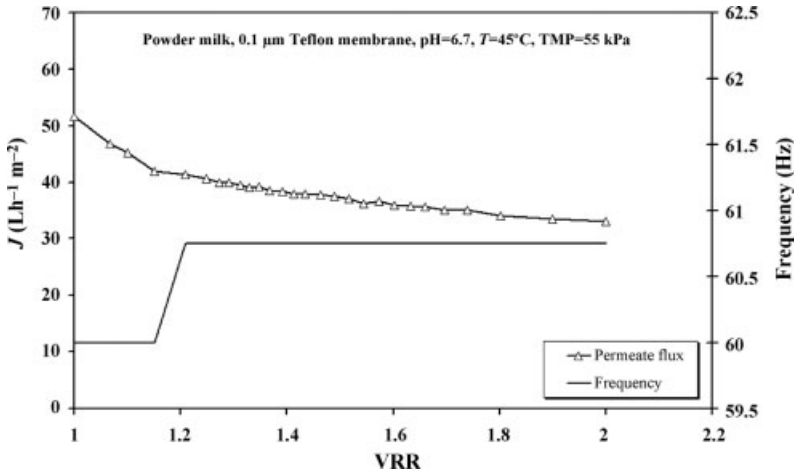

Figure 3.5 Variation of permeate flux and frequency with VRR in MF of powder milk (from Ref. [45] with permission).

rejection. Similar tests, performed with the same VSEP pilot and membrane, but using powder skim milk with same protein composition as pasteurized milk have been reported in [45]. In concentration tests at 55 kPa, the flux decayed from 50 to 33 L h^{-1} m^{-2} at VRR = 2 (Figure 3.5). The faster initial rate of decay is due to the lower frequency of 60.2 Hz. When the 0.1-μm membrane was replaced by a 150-kDa PES one, the permeate flux decayed slowly with increasing concentration from 40 L h^{-1} m^{-2} to 35 at VRR = 2, (Figure 3.6) while permeate turbidity dropped from 160 NTU to about 30 indicating good micelle rejection (Figure 3.6).

When TMP was varied over a cycle, the permeate flux was reversible, but α-La transmission, which was between 70 and 80%, and β-Lg (30–35%) decayed with time

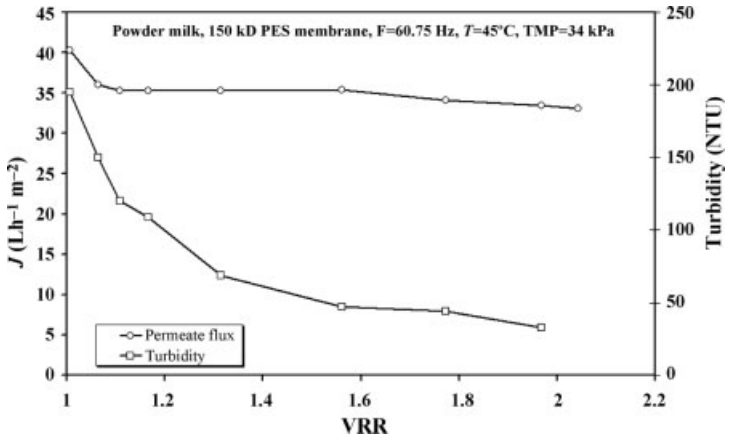

Figure 3.6 Variation of permeate flux and turbidity with VRR in ultrafiltration of powder milk (from Ref. [45] with permission).

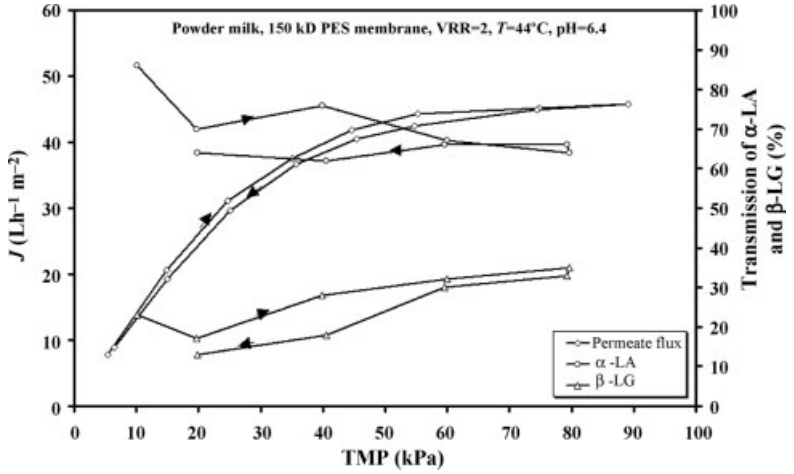

Figure 3.7 Variation of UF permeate flux and whey-protein transmission with TMP during a pressure-variation cycle (from Ref. [45] with permission).

(Figure 3.7) due to internal fouling. Espina *et al.* [46] microfiltered skim UHT milk using a MSD pilot with six 9-cm diameter rotating ceramic membranes with 0.2-μm pores. Permeate fluxes reached a maximum of 120 $L\,h^{-1}\,m^{-2}$ at a rotation speed of 1930 rpm, a TMP of 100 kPa, and 40 °C (Figure 3.8). Permeate turbidity was less than 20 NTU, indicating excellent casein micelles rejection. In concentration tests, the permeate flux decayed logarithmically with VRR (Figure 3.9) according to the thin-film theory of Blatt *et al.* [47].

The reduction from 1930 to 1044 rpm has a large effect on permeate flux. Corresponding α-La and β-Lg transmissions, are shown in Figures 3.10 and 3.11

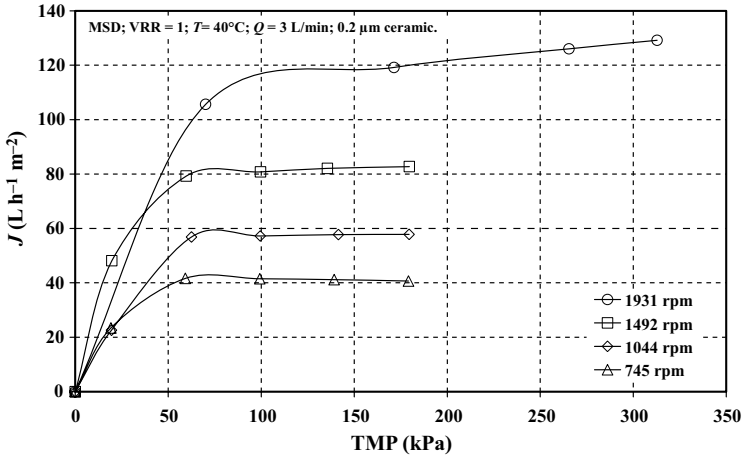

Figure 3.8 Variation of stabilized permeate flux versus TMP with the MSD at different rotation speeds for tests of Figure 3.4 and one at 1930 rpm (from Ref. [46], with permission).

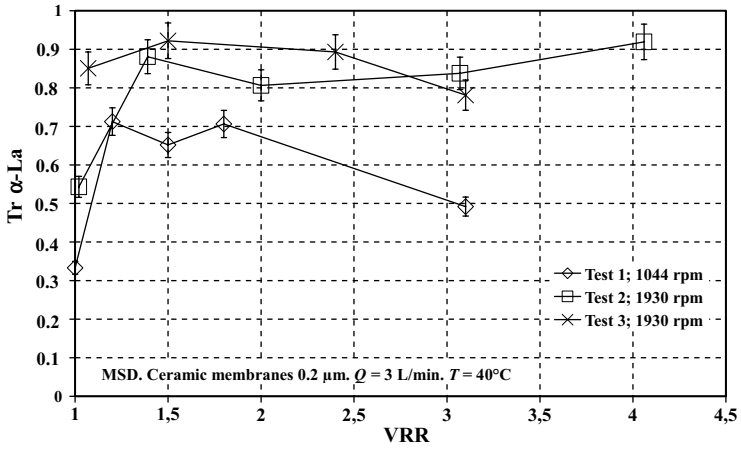

Figure 3.9 Variation of permeate flux with VRR (semi-log) in MF of skim milk with the MSD module for tests 1 to 3 (from ref. [51], with permission).

respectively. At a speed of 1930 rpm, these transmissions remain between 80 and 90% after about 15 min of filtration until the maximum VRR. At 1044 rpm, these transmissions reach a maximum at VRR = 1.3 and decrease at higher VRR to 50% for α-La and 40% for β-Lg. The same group also tested a prototype rotating-disk module, designed at the University of Technology of Compiègne (UTC), consisting in a metal disk equipped with radial vanes rotating at high speed near a fixed 0.15-µm pore PVDF circular membrane. This module yielded higher fluxes, up to 200 L h^{-1} m^{-2} at a speed of 2000 rpm and 200 kPa (Figure 3.12) since the membrane shear rate was higher than in the MSD due to the larger membrane radius (15 cm instead of 9). Permeate turbidity was also very low at 10 NTU, indicating casein rejection higher

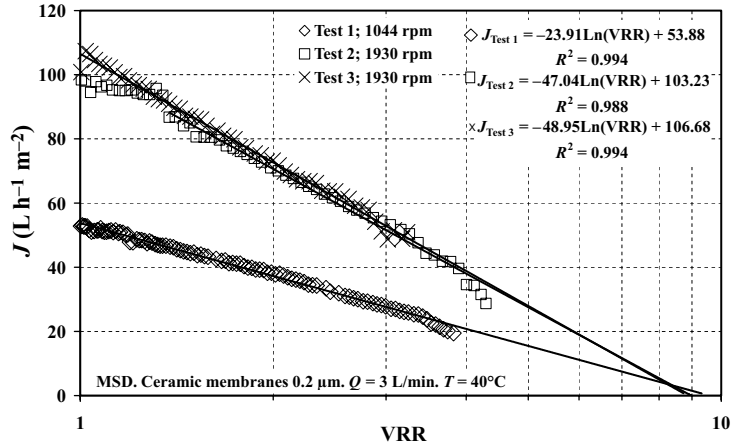

Figure 3.10 α-La transmission with VRR for MF tests of Figure 3.9. (from Ref. [51], with permission).

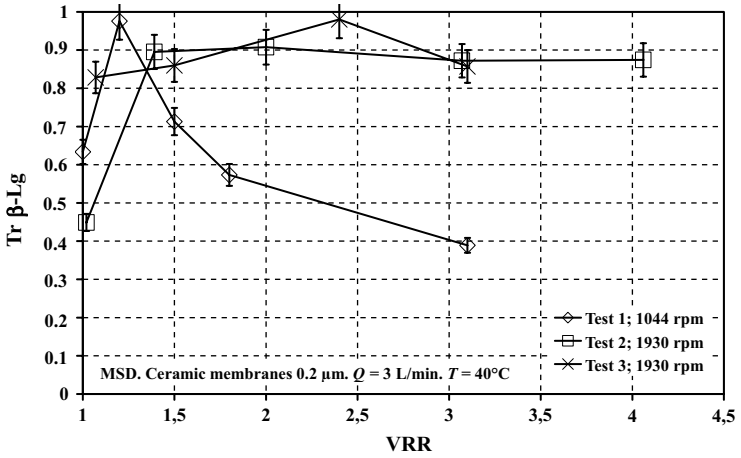

Figure 3.11 β-Lg transmission with VRR for MF tests of Figure 3.10 (from Ref. [51], with permission).

than 99.5%. However, α-La and β-Lg transmissions were low, respectively 30–35% for α-La and 8% for β-Lg (Figure 3.13) due in part to the lower membrane cut-off. These data confirmed the high potential of rotating disks and rotating membrane systems that performed better than the VSEP for this application. As seen in Section 3.3, permeate fluxes with tubular ceramic membranes in UTP mode were generally between 70 and 90 $L\,h^{-1}\,m^{-2}$ at 50 °C with tangential velocities of about 7 $m\,s^{-1}$ and casein micelles rejection was generally not as high as with the MSD. α-La and β-Lg

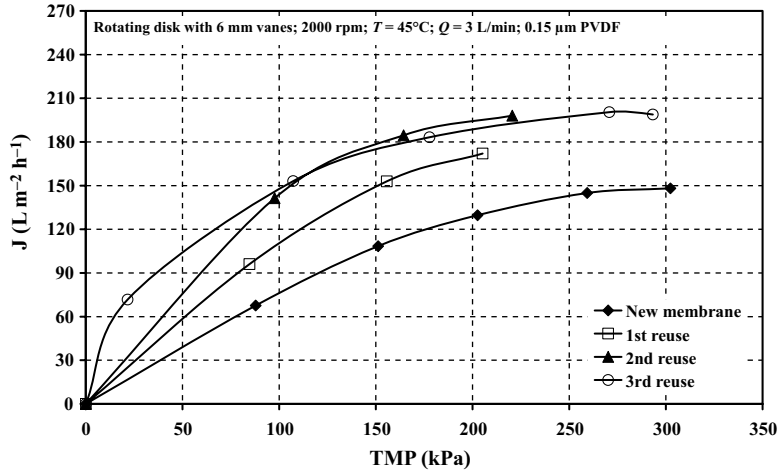

Figure 3.12 Variation of stabilized permeate flux versus TMP using the rotating-disk module with vanes at 2000 rpm and with a new 0.15-µm PVDF membrane and after several reuses (from Ref. [46], with permission).

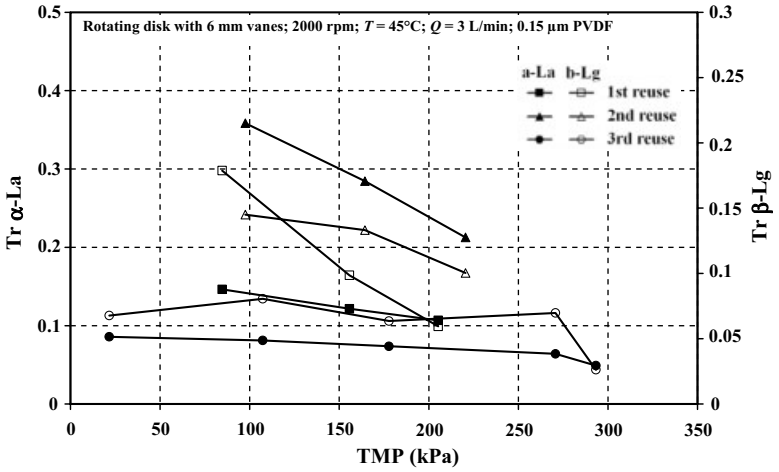

Figure 3.13 Variation of α-La and b-Lg transmissions with TMP for the tests of Figure 3.12 (from Ref. [46], with permission).

transmissions obtained with the MSD pilot compared favorably with those reported in UTP mode [13].

3.3.3.2 Dynamic Ultrafiltration of Skim Milk

Jaffrin *et al.* [48] compared the performance of rotating disk and VSEP modules equipped with the same PES 50-kDa membrane. Two types of disks were tested, a flat (or smooth) disk and a disk equipped with eight 6-mm high radial vanes. Due to reduced concentration polarization by high shear rates, the permeate flux kept rising with increasing TMP for the disk with vanes that produces a maximum shear rate at disk periphery of $2.8 \times 10^5\,s^{-1}$, until at least 600 kPa, reaching $200\,L\,h^{-1}\,m^{-2}$ (Figure 3.14). With the same disk rotating at 1000 rpm, the maximum membrane shear rate fell to $8.2 \times 10^4\,s^{-1}$, which was about the same as for a smooth disk rotating at 2000 rpm. Permeate fluxes for these two cases were almost the same, reaching $115\,L\,h^{-1}\,m^{-2}$ at 400 kPa. The VSEP, which had a slightly higher shear rate of $1.15 \times 10^5\,s^{-1}$, reached the same flux, but at a higher TMP of 850 kPa. The same comparison, but made during concentration tests is shown in Figure 3.15. The highest permeate fluxes were obtained with a disk equipped with vanes rotating at 2000 rpm, which is logical, since it corresponds to the maximum shear rate. The permeate flux decayed slowly, from 130 to $120\,L\,h^{-1}\,m^{-2}$ until VRR = 3, as it is pressure limited. Then it dropped at a faster rate as Ln (VRR^{-1}) as the flux became mass transfer limited at higher VRR, since the increase in viscosity lowered the shear rate. When a smooth disk was used at the same speed, the flux was lower and mass-transfer limited as the membrane shear rate was one third of the previous case. The VSEP permeate flux was slightly lower than for the smooth disk for VRR < 3.5, even though TMP was higher, 400 kPa instead of 300. For VRR > 3.5, however, the VSEP flux exceeded that of the rotating disk, since the VSEP shear rate decreases less at high concentration than with the rotating disk.

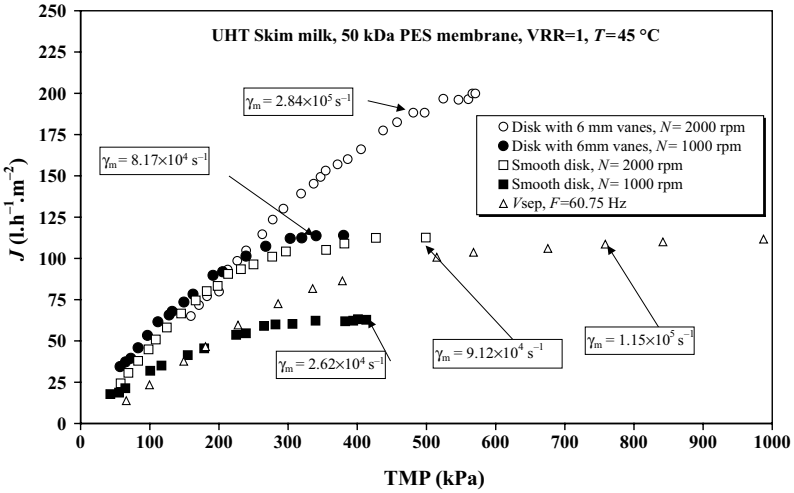

Figure 3.14 Variation of permeate flux in UF of skim milk versus TMP using the rotating-disk module with a smooth disk and a disk with vanes, and the VSEP (from ref. [48], with permission).

Ding *et al.* [49] ultrafiltered UHT milk with the same PES 50 kDa membrane as in [48] using a rotating-disk module. They measured the net power (P_N) consumed by friction on the disk as function of rotation speed (N) together with corresponding permeate flow rates Q_F (Figure 3.16). Since in a small pilot, the power consumed by the shaft and internal parts of motor is disproportionably high, the power consumed by the motor with an empty module was subtracted from the power measured at the same speed during milk filtration, in order to obtain power consumed by disk friction

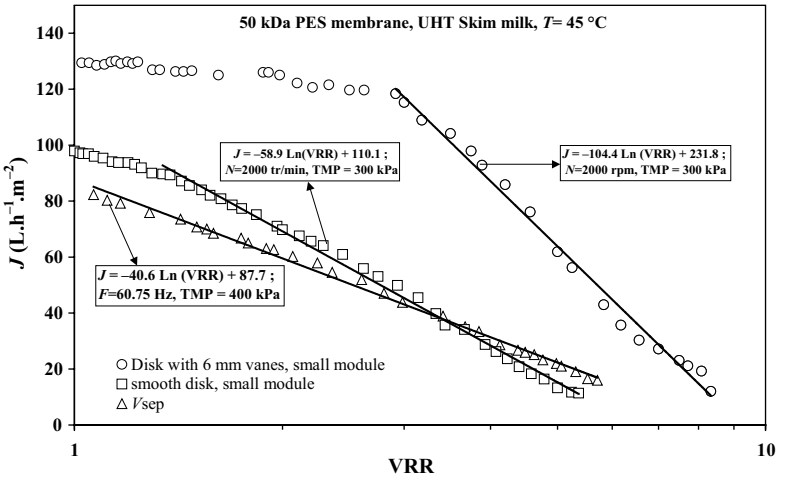

Figure 3.15 Variation of permeate flux in UF of skim milk versus VRR in semi-logarithmic scale using the rotating-disk module with two types of disks and the VSEP (from ref. [48], with permission).

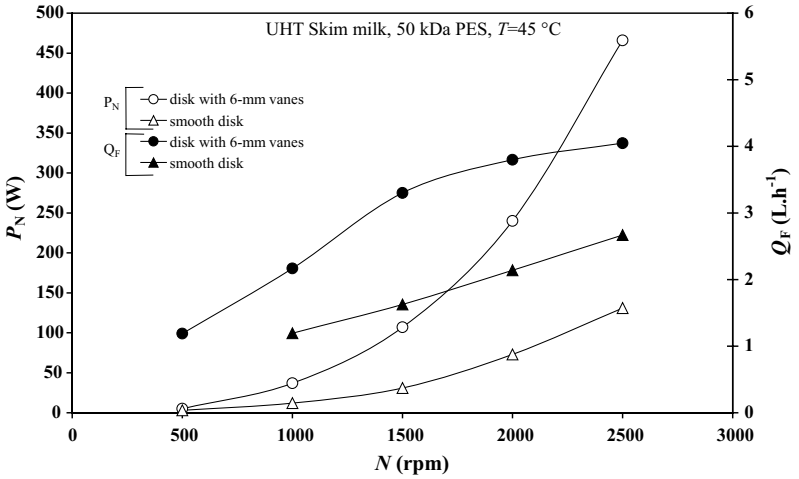

Figure 3.16 Variation of net power consumed by the disk P_N and permeate flow rate Q_F for two types of disks with rotation speed (from ref. [49], with permission).

alone, which will be the dominant part in a large module. As expected, the power increased as N^2 and was larger for a disk with vanes and the gap between the two disks widened at large speed. The specific power per m^3 of permeate, plotted in Figure 3.17, which is given by the ratio P_N/Q_F, increased with N and was higher for a disk equipped with vanes than for a smooth one, as the increment in permeate flow rate with vanes was less than the power increase. But vanes increase the flux and permit to lower membrane area. Thus, higher energy costs may be offset by a reduction in

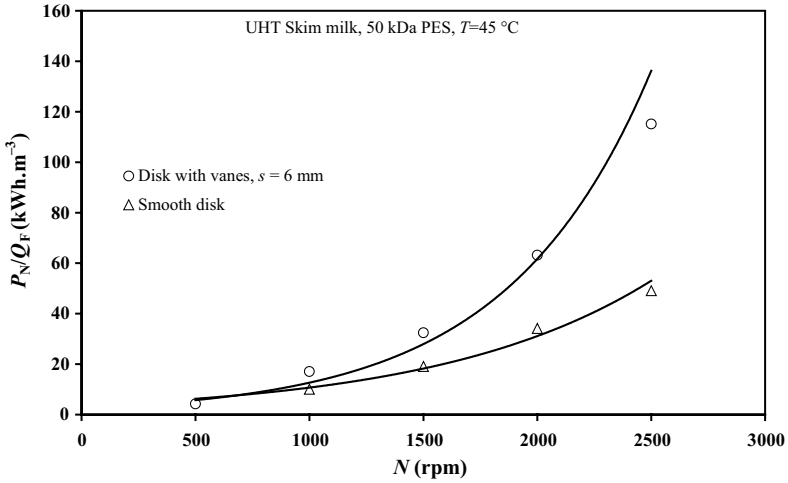

Figure 3.17 Variation of specific energy consumed by the disk per m^3 of permeate using data of (from ref. [49], with permission).

equipment cost. Optimal configuration and rotation speed may be determined from appropriate financial and economic information.

3.3.3.3 Total Protein Concentration by UF for Cheese Manufacturing

Akoum *et al.* [50] used a VSEP pilot equipped with a 10-kDa PES membrane permitting high protein rejection to concentrate caseins and whey proteins from a powder low-heat skim milk with the same composition as fresh milk. Permeate fluxes obtained at 46 °C and initial concentration are given in Figure 3.18 for various vibration frequencies. In order to reduce wear and maintenance, the VSEP is not used at its maximum frequency in normal industrial use, but with a membrane displacement amplitude of 2–2.5 cm at the rim, rather than at the maximum of 3 cm at resonance. This corresponds to frequencies of 60–60.2 Hz for this pilot. The permeate flux kept increasing with TMP until 1500 kPa, even at 60 Hz where it reached 70 $L\,h^{-1}\,m^{-2}$, while at lower frequencies the maximum was reached at 600 kPa or less. Variations of permeate fluxes in concentration tests without permeate recycling are displayed in Figure 3.19 and decay linearly with increasing Ln(VRR). The maximum theoretical VRR, extrapolated to zero flux, was about 17 for all frequencies, thus, higher than corresponding values obtained with cross-flow filtration, which are less than 10.

A comparison of variation of permeate fluxes versus milk dry mass in %, which is proportional to the concentration factor, is shown in Figure 3.20 for UHT and powder skim milks and for 10- and 50-kDa membranes. Data for powder and UHT milks are very close although, in UHT milk, whey proteins are partially denatured and the flux dropped a little faster with increasing concentration for the 50-kDa membrane, due perhaps to larger internal fouling.

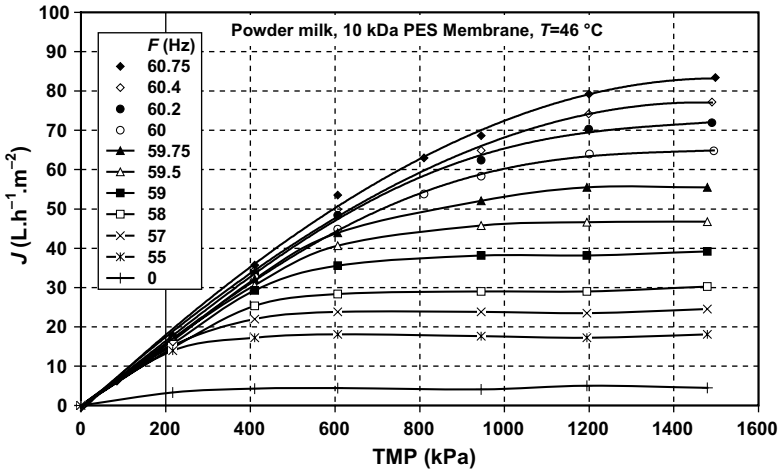

Figure 3.18 Variation of permeate flux in UF at 10 kDa of skim milk with TMP using a VSEP at various frequencies and VRR = 1 (from ref. [50], with permission).

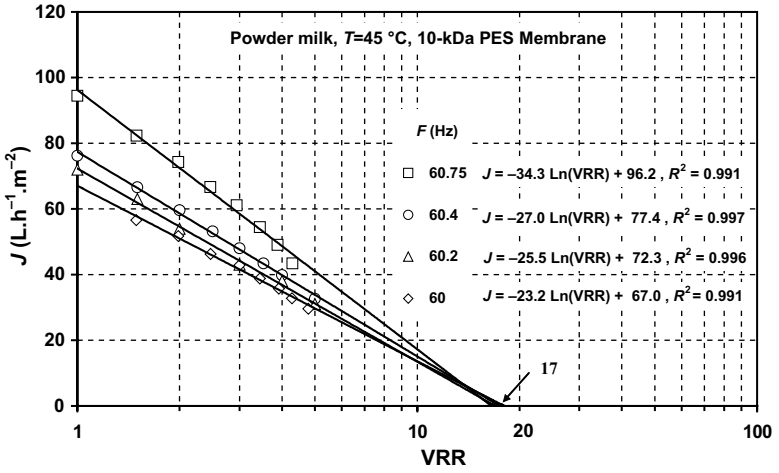

Figure 3.19 Variation of permeate flux in UF at 10 kDa of skim milk with VRR using a VSEP at various frequencies and a TMP of 1.5 MPa (from ref. [50], with permission).

3.3.3.4 α-La and β-Lg Protein Fractionation by UF

Espina *et al.* [51] used a UTC rotating-disk module equipped with a 50-kDa PES membrane on skim UHT milk permeate obtained after MF at 0.2 μm with ceramic membranes to separate α-La in UF permeate from β-Lg in retentate. The UF permeate flux obtained at 40 °C, shown in Figure 3.21 was higher, at 200 L h^{-1} m^{-2} and VRR = 4, than those reported in Section 3.2 with cross-flow UF, which were less than 100 L h^{-1} m^{-2}. α-La transmission, shown in Figure 3.22, rose from a minimum of 11% at VRR = 1.3–24% at the maximum VRR of the test (3.1). β-Lg transmission

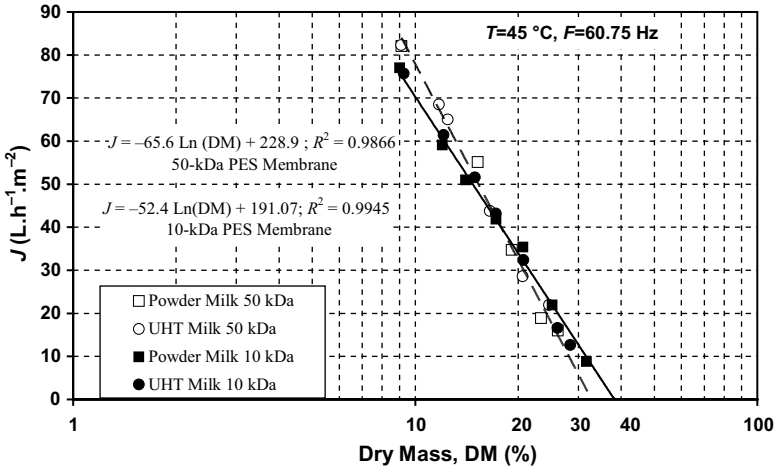

Figure 3.20 Variation of permeate flux in UF at 10 and 50 kDa of UHT and powder skim milks with dry mass percentage using a VSEP (from ref. [50], with permission).

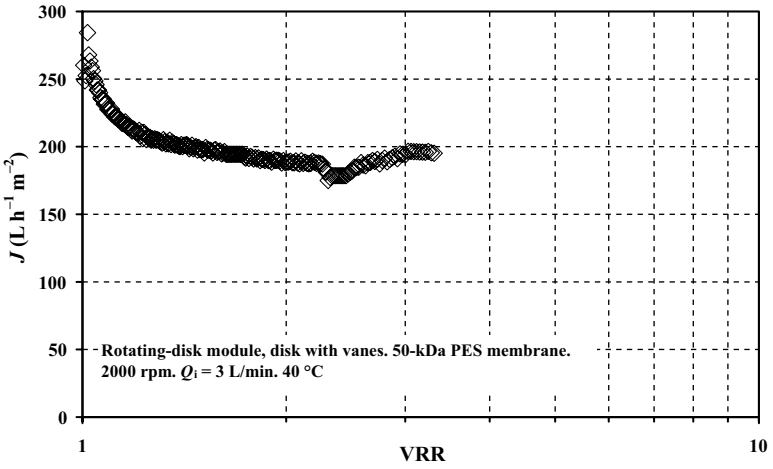

Figure 3.21 Variation of permeate flux with VRR for UF at 50 kDa of UHT milk MF permeate using the rotating-disk module at 2000 rpm (from ref. [51], with permission).

dropped to about 2–3% at VRR > 1.8, so that selectivity (Tr_α/Tr_β), also shown in Figure 3.22, rose to 8 at VRR = 3.1. This selectivity was close to that of 10.5 obtained with a 50 kDa membrane, but on a binary protein mixture by Cheang and Zydney [26], after optimizing ionic force and pH. By contrast, Gésan-Guiziou *et al.* [52] obtained a transmission of only 9% for α-La and 6% for β-Lg during the ultrafiltration of redissolved precipitate from Gouda whey protein concentrate with a 50-kDa Carbosep membrane, at VRR = 10 and 50 °C.

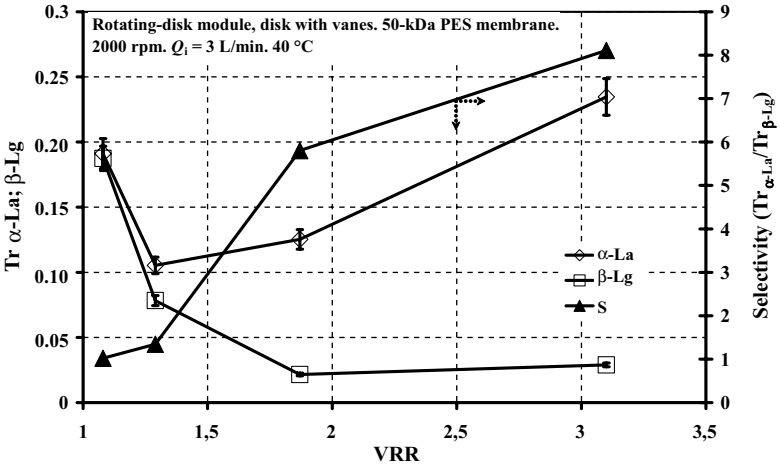

Figure 3.22 α-La and β-Lg transmissions, and variation of selectivity (Tr α-La)/(Tr β-Lg) versus VRR for test of Figure 3.21 (from ref. [51], with permission).

Bhattacharjee *et al.* [53] separated β-Lg from whey protein concentrate obtained from raw casein whey by centrifugation followed by a MF at 0.45 μm. Their dynamic filtration module consisted in a circular polymer membrane of 76 mm diameter rotating inside a cylinder, near a disk stirrer rotating in the opposite direction at 500 rpm. They used a complex three-stage process, starting with a diafiltration at 5 kDa to remove lactose, minerals and salts. The retentate was then ultrafiltered at 30 kDa, after addition of hydrochloric acid to lower the pH to 2.8 in order to obtain monomer β-Lg and α-La, while bovine serum albumin, lactoferrin and immunoglobulins were collected in retentate. When the membrane was at rest, the flux decayed from 200 L h to 20 $L\,h^{-1}\,m^{-2}$ after 20 min of filtration. When the membrane speed was set to 300 and 600 rpm, the flux stabilized to 100 and 115 $L\,h^{-1}\,m^{-2}$, respectively. The final separation between monomer β-Lg and α-La was obtained by ion-exchange membrane chromatography as the molecular weights of these two proteins were too close to be separated by UF. The separation factor between β-Lg and α-La increased with the pH of the loading buffer in ion–exchange chromatography to reach a maximum of 4.7 at pH = 5.0. The final purity of β-Lg, relative to total proteins, was 0.87. The lowering of pH to 2.8 permitted to increase the β-Lg/α-La ratio to 17.15 as compared to 9.64 when β-Lg remained in dimer form at pH = 5.6.

3.3.4
Treatment of Dairy-Process Waters by Dynamic NF and RO

Akoum *et al.* [54] used a L101 VSEP pilot to treat "white" waters represented by one volume of skim UHT milk diluted with two volumes of pure water. The initial COD of this diluted milk, mainly due to lactose, was 36 000 $mgO_2\,L^{-1}$, which corresponds to a highly charged effluent. The VSEP was equipped with the same Desal 5DK and 5DL as spiral-wound modules used by Balannec *et al.* [34]. Variations of permeate flux, COD and conductivity (proportional to ion concentration) obtained using the VSEP at 25 and 45 °C are represented in Figure 3.23 as a function of TMP for a 5DL membrane and initial concentration. For comparison, the graph also indicates permeate flux and COD provided by the spiral module equipped with the same 5 DL membrane at 25 °C and 1.9 MPa. The spiral module flux was 24 $L\,h^{-1}\,m^{-2}$ or one third of the VSEP flux at same TMP and temperature (72 $L\,h^{-1}\,m^{-2}$). The spiral module COD was 128 $mgO_2\,L^{-1}$, five times higher than the VSEP COD (24 $mgO_2\,L^{-1}$) under the same conditions. It is interesting that the high shear rates of the VSEP, not only increase significantly the permeate flux as compared to cross-flow filtration, but decrease lactose and ions transmission, responsible for permeate COD in NF by reducing their concentration at the membrane due to lower concentration polarization. In concentration tests without permeate recycling (Figure 3.24), the VSEP retains its high performance with a permeate flux which decayed linearly with increasing VRR to 30 $L\,h^{-1}\,m^{-2}$ at VRR = 5, 1.9 MPa and 25 °C, against 11 $L\,h^{-1}\,m^{-2}$ for the spiral-wound module under same conditions. Presumably, the flux difference between the two modules would have been larger at higher TMP as the VSEP flux kept increasing until TMP = 4 MPa, while the spiral-module one leveled off at about 2 MPa. However,

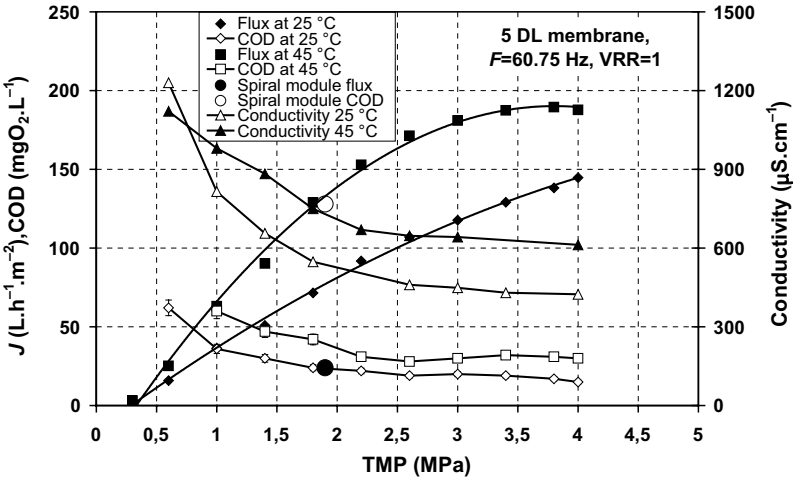

Figure 3.23 Variation of permeate flux, permeate COD and conductivity of diluted milk with TMP using a VSEP and a nanofiltration membrane and comparison at 25 °C with a spiral-wound module with the same membrane and conditions (from ref. [54], with permission).

VSEP COD, which was half that of spiral module up to VRR = 2, increased faster at high VRR and COD of both modules reached 350 $mgO_2\,L^{-1}$at VRR = 5.

A similar investigation, but using a rotating-disk module, was carried out with the same model effluent (diluted milk) and a Desal 5 DK membrane at a temperature of 45 °C and TMP of 4 MPa by Frappart *et al.* [55]. Variations of permeate flux with VRR at rotation speeds of 1000 and 2000 rpm and two types of disks are presented in Figure 3.25. As expected, the highest permeate fluxes were obtained with a disk equipped with 6-mm radial vanes and rotating at 2000 rpm, producing a maximum

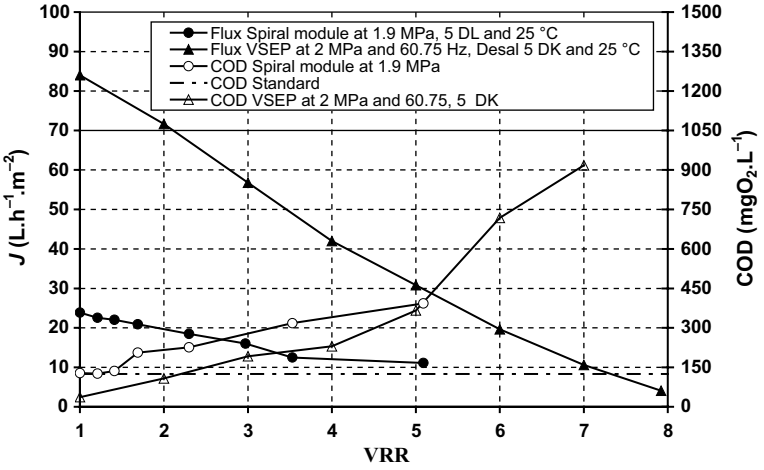

Figure 3.24 Comparison of permeate flux and COD variations with VRR in NF. (from ref. [54], with permission)

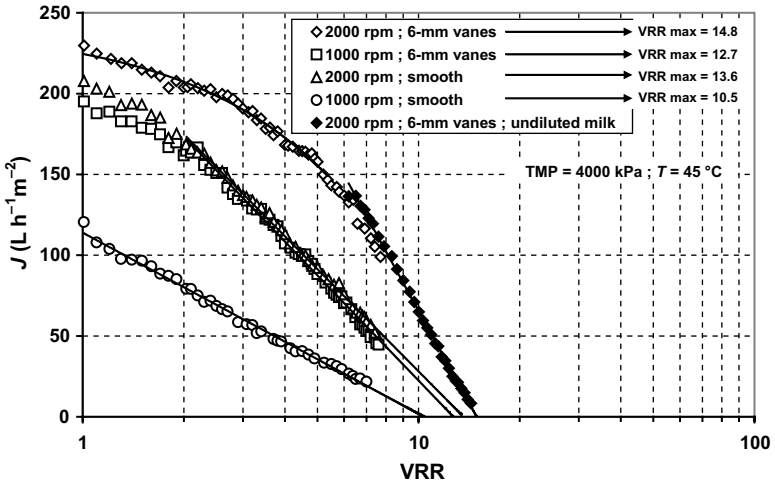

Figure 3.25 Variation of permeate flux with VRR at a TMP of 4 MPa using a rotating-disk module at 1000 and 2000 rpm for two types of disk and a 5DK NF membrane (from ref. [55], with permission).

shear rate at membrane periphery of $4.4 \times 10^{-5}\,s^{-1}$. This flux decayed from 225 L $h^{-1}\,m^{-2}$ at VRR = 1 to 140 at VRR = 5, while with the same disk rotating at 1000 rpm or with a smooth disk rotating at 2000 rpm, the flux at VRR = 5 dropped to about 90 L $h^{-1}\,m^{-2}$ as respective shear rates were only 1.2×10^{-5} and $1.1 \times 10^{-5}\,s^{-1}$. Corresponding variations of permeate COD with VRR are represented in Figure 3.26. These COD are lowest at the highest shear rates, but they exceed the allowed limit (rejection standard) of 125 $mgO_2\,L^{-1}$ above VRR = 4, so that a RO step may be

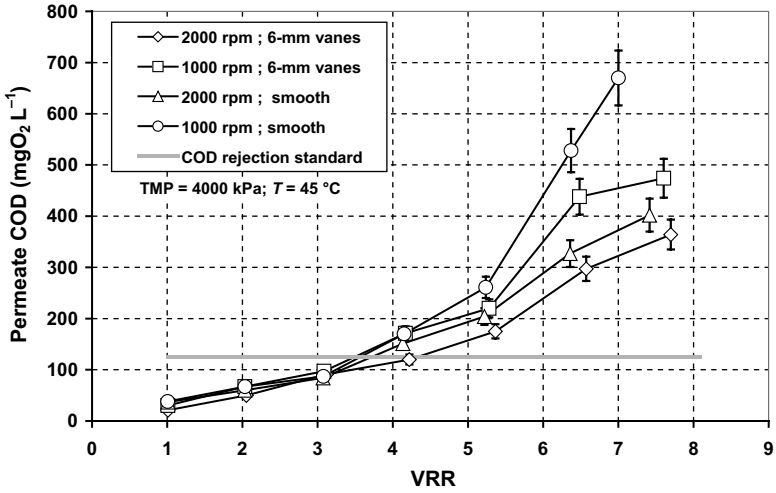

Figure 3.26 Variations of permeate COD with VRR for the tests of Figure 3.25 (from ref. [55], with permission).

necessary at high VRR. However, for usual dairy effluents with lower initial COD, the limit may be respected with a single NF step.

3.4 Conclusion

The use of membrane processes in the dairy industry has increased significantly during the last 20 years. Bacteria removal by MF avoids serum protein denaturation and nutritional losses due to UHT or pasteurization treatment. Recently available Isoflux membranes with permeability gradient are replacing UTP processes with cocurrent permeate recirculation that required more energy. Membrane processes for protein fractionation are emerging as they can be extrapolated to large volumes and automatized production, unlike ion exchange, affinity chromatography and selective precipitation. According to Brans *et al.* [3], their technical advantages should spur their industrial development. But milk is a complex fluid that presents a challenge to membrane processes, as many of its components induce fouling, which requires use of large fluid velocities and highly selective membranes. Thus, process conditions and fouling control methods must be further optimized.

Dynamic filtration, which has clearly proved its efficiency to reduce membrane fouling in MF and concentration polarization in UF, NF and RO, may play an important role, especially for extracting valuable milk components. Systems with rotating ceramic membranes and vibrating ones seem well suited for this application, but their costs are presently higher than those with tubular membranes, because of their small production. But their cost should decrease as sales increase. In addition, dynamic filtration gives the choice between using high shear rates with large rotation speeds in order to increase permeate flux, or to use moderate rotation speeds giving the same flux as in cross-flow filtration, but with a lower energy per m^3 of permeate. Thus energy savings may compensate the higher initial cost. Dynamic microfiltration with rotating ceramic disks may be another alternative to cocurrent permeate recirculation.

Concerning applications involving NF and RO, the industrial future of dynamic filtration is more delicate to predict. This chapter has clearly shown the high performance of VSEP and rotating-disk modules, both equipped with polymer membranes, as no NF and RO ceramic disks seem to be yet available. As said earlier, polymeric-membrane modules for dynamic filtration are more complex and costlier to build than ceramic membranes modules. In addition, large spiral-wound modules, which are built in large quantities for water desalination and water treatment are very inexpensive, about 10–15 € per m^2 and are also very compact. So dynamic modules probably cannot presently compete with spiral-wound NF and RO modules in terms of cost per m^3 of permeate, even if their membrane area is much smaller for the same output. The situation may be different for fractionation applications that are generally carried out with tubular ceramic membranes of much higher cost than spiral-wound modules, and for which a high selectivity and a low energy consumption are important.

Acknowledgments

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Abbreviations

DF	diafiltration
MF	microfiltration
NF	nanofiltration
RO	reverse osmosis
TMP	transmembrane pressure
UF	ultrafiltration
UHT	ultrahigh temperature
UTC	University of Compiègne
UTP	uniform transmembrane pressure
VRR	volume-reduction ratio

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