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Classical and Recent Applications of Membrane Processes in the Food Industry

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Abstract
Membrane processes are extensively used in the food industry, especially in the dairy, wine and beer, fruit juice, and sugar industries. In the dairy industry, removal of bacteria and spores from skim milk (cold pasteurization), separation of casein micelles, separation and fractionation of fat globules from whole milk, concentration and demineralization of whey and milk ultrafiltration permeate, fractionation of whey proteins, and desalination of whey membrane processes are realized by membrane processes. In the wine and beer industry, cross-flow of wines and beer clarification and stabilization of wines by electrodialysis are of common use. Fruit juice processing involves clarification, concentration, and deacidification. In the sugar industry, purification and demineralization are realized by membrane processes. This review presents these membrane processes by focusing on the main parameters and phenomena like permeation flux, selectivity, and fouling of membranes. Innovative pilot designs and cleaning methods are presented. The potentials of more unusual techniques like membrane emulsification and electrodialysis of organic acids from fermentation broth are also underlined. A specific attention is paid to the recent development of membrane processes in the food industries.

Keywords Membrane · Process · Industry · Food · Milk · Fruit juice

Introduction

Membrane processes have become major techniques in the food industry over the last decades. The dairy industry, followed by beverages (wine, beer, fruit juices, etc.) and sugar products are the main fields of applications ([11, 18, 44, 127]). Common membrane processes are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) reverse osmosis (RO), and electrodialysis (ED).

This review focuses on classical applications and their evolution during the two last decades. Previous books and reviews on the topic of membrane processes for food applications are first listed. The following sections focus on membrane processes used in the dairy, wine and beer, fruit juice, and sugar industries. A particular attention is devoted to parameters which control these techniques, to membrane fouling, as well as to industrial pilot plants which are currently in use. Recent trends (new processes, new configurations, new products, etc.) are underlined. A final section presents some processes which may find their way in the food industry, including ED of organic acids from fermentation broth and membrane emulsification.

Membrane Processes

This section gives some very general background on membrane processes. They can be found in several books (for example, [30, 51, 52, 148]) that will not be cited later.

Membranes are usually classified accordingly to their average pore sizes. Membranes are considered to be dense when the transport of components involves a stage of dissolution and diffusion across the material constituting the membrane. MF membranes typically have pore sizes on the order of 0.1 to 10 μm. UF membranes have pore sizes in the range of 0.001 to 0.1 μm and are capable of retaining species in the molecular weight range of 300–10^6 Da. RO membranes retain solutes with molar mass below 1000 Da, and NF membranes retain solutes in the range of molar mass between 1000 and 3000 Da.

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The two standard modes of membrane operation are dead-end and cross-flow. In the dead-end mode, the fluid to be filtered is forced through the membrane pores usually by applying a pressure on the feed side. In cross-flow mode, the feed flows parallel to the membrane surface and permeates through the membrane due to a pressure difference. The cross-flow reduces the formation of the filter cake to keep it at a low level. In cross-flow mode, the most commonly used membrane modules include hollow fiber, tubular, flat plate, and spiral wound devices. Hollow fiber and spiral wound modules have the highest membrane packing densities since they have the thinnest channels. However, this makes them more susceptible to fouling and can make cleaning more difficult [148]. Any of these cross-flow devices can be used in dead-end; however, these configurations are susceptible to high fouling [148].

Several parameters are used to characterize membranes, devices, and processes. The transmembrane pressure (TMP) is the difference between the pressures on the feed side and on the permeate side. For a cross-flow device, the pressure on the feed side is calculated as the mean of the pressures at the inlet and outlet of the device. MF membranes are characterized by their mean pore size and UF and NF membranes by their molecular weight cutoff (MWCO) defined as the molecular weight above which molecules are at least 90% rejected by the membrane.

The apparent sieving coefficient, S_a, represents the transmission of a partially rejected species through the membrane:

$$S_a = \frac{C_p}{C_b} \tag{1}$$

where C_p and C_b are respectively the species concentrations in the permeate stream and in the feed (or bulk) stream, respectively. The apparent rejection coefficient is defined as follows:

$$R_a = 1 - \frac{C_p}{C_b} \tag{2}$$

The efficiency of binary species fractionation is commonly expressed in terms of the selectivity, ψ:

$$\psi = \frac{S_{a1}}{S_{a2}} \tag{3}$$

where subscript 1 stands for the preferentially transmitted species and subscript 2 for the preferentially retained species.

In almost all membrane processes, performances (permeate flux, membrane selectivity, etc.) are limited by two distinct phenomena: concentration polarization and fouling. These phenomena are well recognized and described in the above reference text books and numerous articles. Also, recent articles emphasized the role of concentration polarization and fouling in membrane processes [35, 103, 116, 141, 142]. Concentration polarization is due to the selective transport of some species through the membrane and the accumulation of other species near the membrane surface. This results in a decrease of permeate flux. In general, concentration polarization is a reversible phenomenon in that sense that species will diffuse back towards the feed by adjusting the operating conditions for example by increasing the cross-flow velocity. In addition, concentration polarization may be associated to the formation of a gel layer at high species concentrations which is not reversible by simply changing the operating conditions. The formation of a gel layer requires cleaning to restore the membrane properties [35, 103, 141, 142]. In contrast to concentration polarization, fouling is the irreversible alteration of the membrane caused by physical and/or chemical interactions between the membrane and the species present in the feed solutions or suspensions. Fouling results in a complete or partial pore blockage and/or formation of a deposit on the membrane surface, depending on the properties of retained species, membranes, operating conditions, etc. Like concentration polarization, fouling manifests itself as a decrease in permeate flux and/or membrane selectivity. This alteration requires cleaning or replacement of the membrane. Membrane fouling has been extensively studied. Recent trends include the in situ real-time monitoring techniques of membrane fouling, with advanced characterization techniques like HPLC coupled mass spectrometry, and advanced simulations methodology like molecular simulation [35, 116].

Membrane cleaning is an essential component of almost all membrane processes. Membrane cleaning is detailed in the above reference text books, and in many articles, it is also discussed in the following recent articles [35, 103, 141, 142]. Concentration polarization and fouling occur during operation, causing the permeate flux to drop below a certain acceptable level. The gel layer and/or foulants must then be removed from the membrane using an appropriate cleaning procedure. Cleaning has a significant impact on process operations and the commercial viability of the process. It is accomplished by physically removing the gel layer and/or foulants, for example by backflushing, and/or by using a specific cleaning solution containing appropriate detergents and/or chemicals. The cleaning treatment must effectively remove and/or dissolve the gel layer and/or foulants while not exceeding the mechanical or chemical limits of the membrane.

Previous Reviews

Several books and general reviews are available on membrane processes for food applications. A recent book is specifically devoted to membrane processing for dairy and

beverage applications [127]. A large range of processes are covered including processing of liquid milk, fermented milk, cheese, whey, concentrated milk and powders, fruit juice, beer and cider, and wine and vinegar. In another book [108] described membrane technologies for food applications, including dairy fractionation, ED, pressure-driven membrane processes in alcoholic beverages, membrane emulsification, contactors and bioreactors, as well as membranes for food packaging. The book edited by Cassano and Drioli [26] describes specifically integrated membrane operations in the food production. Integrated membrane operations are discussed in different areas such as fruit juice processing, milk and whey processing, winemaking, brewing and sugar production, purification of soy extract, and bioactive compounds. Applications of ED are also presented as well as emerging membrane processes, such as biocatalytic membrane reactors and membrane emulsification.

Other reviews focus on more specific aspects. For example [16] reviewed ED-based techniques in the food industry, including dairy, sugar, beverage, wine, and organic acid industries. ED-based processes are used to concentrate, purify, or modify foods. The low-energy consumption, modular design, efficiency, and ease of operation, as well as the low impact on heat sensitive food products, are some of the advantages of ED. In addition, ED differs from pressure-based membrane process (UF, NF, RO), as it does not separate particles according to their size, but to their electrical charges.

[44] gave a complete overview of applications in the dairy industry (whey protein concentration, milk protein standardization) and beverage industry (wine, beer, fruit juices). Several industrial plants are described with membrane and module properties (membrane pore size, geometry, membrane area) and parameters of operation (TMP, permeate flux, etc.). [21] summarized the state of the art and challenges of membrane fractionation of milk, including membrane processes for the isolation of serum proteins from whey, the reduction of bacteria and spores in skimmed milk, the separation and/or fractionation of cream, and the concentration of casein micelles. [12] focused on membrane separation processes applied to whey, including the recovery of the main whey components. The authors discussed the adding value of the membrane processes compared with traditional techniques as well as their environmental impact. Other recent reviews focus on membrane separation processes applied to skim milk and whey processing with special attention paid to fouling mechanism analysis that causes flux and selectivity decline [103, 145].

[81] reviewed specifically membrane processes for the concentration of fruit juices, including RO, direct osmosis (DO), membrane distillation (MD), osmotic distillation (OD), and integrated membrane processes. A specific attention is devoted to advantages and disadvantages of each process. [18] discussed literature data published in the years (2000–2017) by focusing on fruit juice clarification and concentration by MF, UF, NF, RO, and membrane distillation (MD). [56] focused their review on cross-flow MF applied to oenology and [135] to beer clarification using membranes with a specific attention paid to fouling.

The following sections give an idea of classical applications and developments of membrane processes in the dairy industry, wine, beer, fruits juice, and sugar industries. They are summarized in Figs. 1 and 2, for classical and more recent applications, respectively. Two less traditional processes are presented in the last section: ED for organic acid recovery from fermentation broth and membrane emulsification for the preparation of food emulsions.

Dairy Industry

Milk is an emulsion of fat globules in an aqueous phase. The aqueous phase consists of suspended and dissolved components, such as casein micelles, serum proteins, lactose, and salts. A typical composition of milk is given in Table 1. The milk composition varies per cow (race, age, stadium of lactation) and depends on the season, climate, and feed [21]. Membrane processes, alongside classical processes, are used in dairy industry for applications such as separation and/or fractionation of fat globules from whole milk, removal of bacteria and spores from skim milk (cold pasteurization), separation of casein micelles from soluble proteins contained in skimmed milk, concentration of casein micelles, and recovery of serum proteins from cheese whey [, 12, 21, 86].

Removal of Bacteria and Spores from Skim Milk (Cold Pasteurization)

Collected milks by the dairy plant present the risk of containing pathogenic bacteria for human such as *Listeria*, *Brucella*, *Mycobacterium*, or *Salmonella* [117]. Therefore, the reduction of bacteria and spores has to be achieved, without changing the functionality of the milk proteins, especially when the milk has to be used for cheese production. The growth of unwanted bacteria and spores can spoil the cheese by a late blowing during ripening. This happens especially for the semi hard and hard cheese types where a combination of high moisture, low salt content, and elevated ripening temperatures provides an environment that allows the spores to develop and generate gas.

Milk centrifugation, called bactofugation, is a possible method for reducing the number of bacteria and spores [21, 114, 117]. This technique, however, is energy consuming, and only 90–95% of the spores are removed by one step of bactofugation. Heat treatments such as high-temperature,

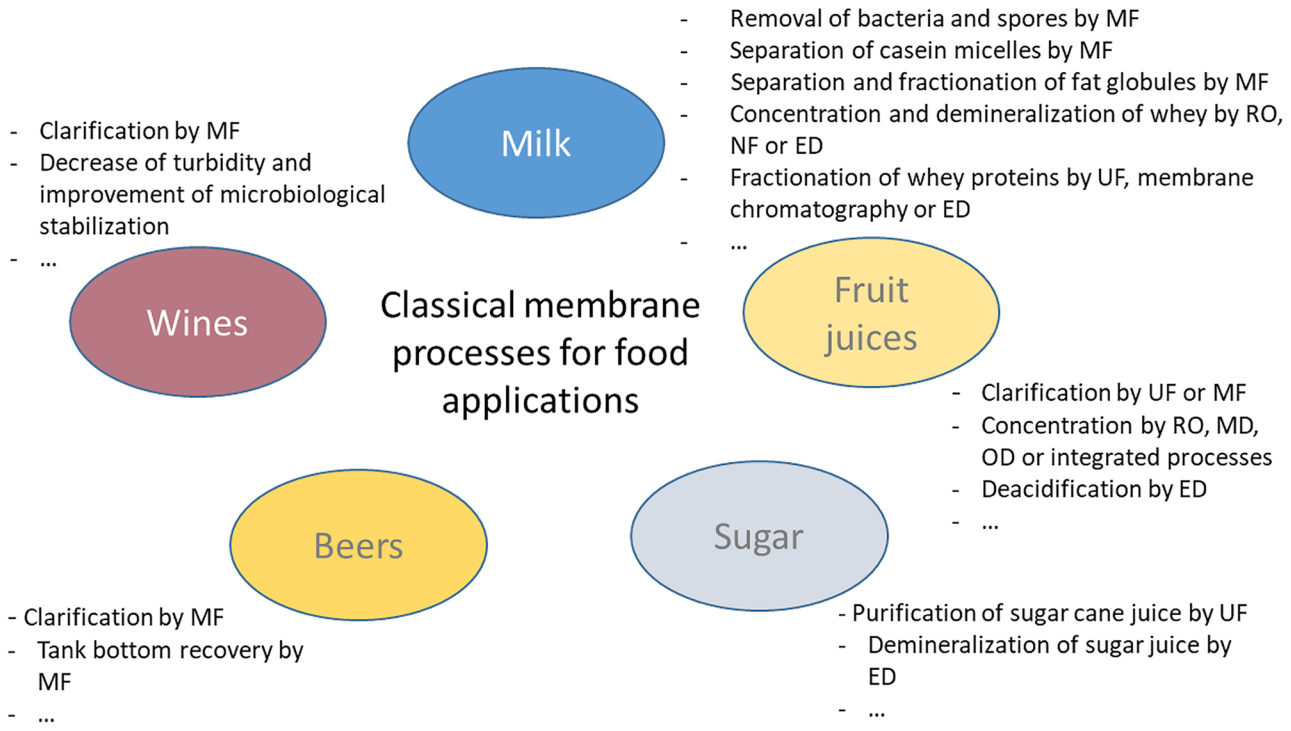

Fig. 1 Classical membrane processes for food applications

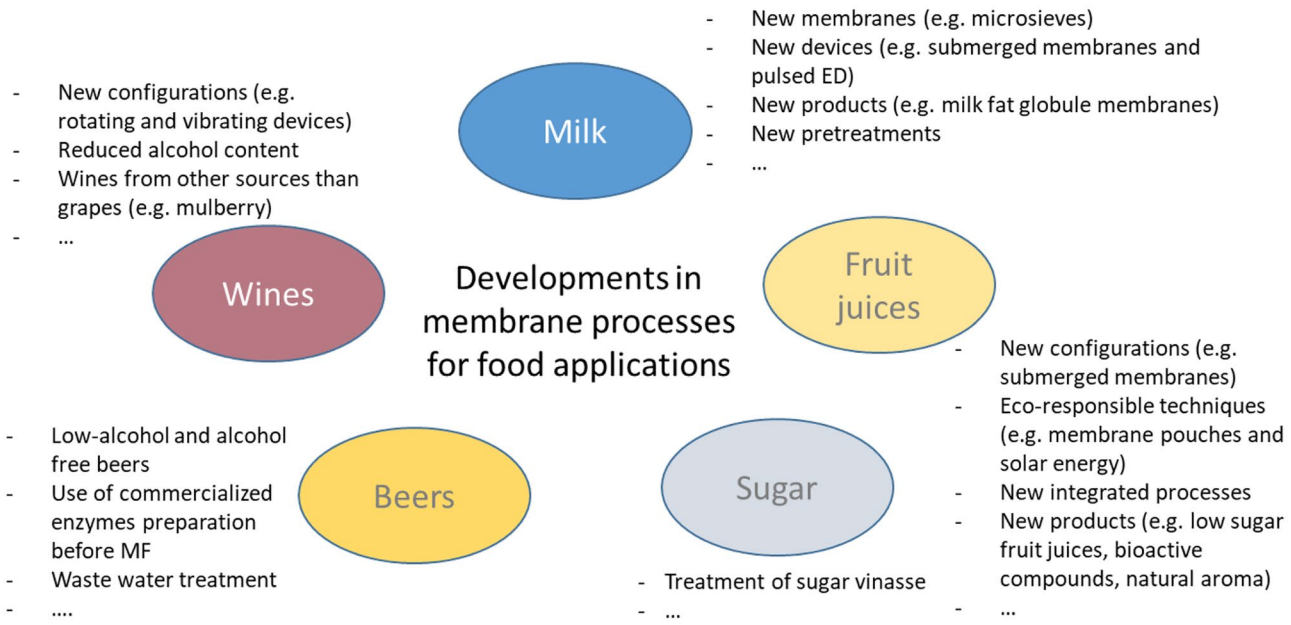

Fig. 2 Development in membrane processes for food applications

short time (HTST) pasteurization, or ultrahigh temperature (UHT) have been applied for more than 50 years and have been proved effective for killing most of the microbial flora. However, the dead cells remain in the milk with potentially active enzymes that will cause alterations of liquid milks during storage, thus reducing shelf life. MF is now largely applied in the dairy industry for bacteria and spore removal. MF reduces the amount of bacteria and spores without

Table 1 Average composition of cow milk: concentration and size distribution (adapted from [21])

	Concentration in whole milk (g/L)	Size range and average (at weight average
Water	87.1	
Fat globules	4.0	0.1–15 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-lactalbumin	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		
Lactose	4.6	0.35 kDa
Mineral substances	0.7	
Organic acids	0.17	
Other		

affecting the taste of the milk and provides longer shelf life than pasteurization. Decimal reduction factors by MF are higher than for bactofugation. Typical values are in the range of 4 [117]. Besides the production of milk with extended shelf life, this technique can be used as pretreatment of skim milk for the production of raw milk cheeses and the reduction of spores in acid cheese milk [21]. The technique is well established. To our knowledge, few recent articles are available on this topic and are related to very specific aspects. For example [72] demonstrated that spore size and surface property may affect the spore retention and that a membrane with pore size of 1.2 μm, instead of the traditional 1.4 μm pore size chosen for this application, could be preferable. However, this smaller pore size can lead to higher retention of casein micelles which causes a decrease in milk protein concentration and also higher membrane fouling.

A commercial MF process for the reduction of bacteria and spores from milk is available under the name Bactocatch (Tetra Laval Co.), initially described in the patent of [78]. Nowadays, skim milk heated to 50 °C is circulated at a velocity of 7.2 m/s along a Sterilox® membrane with an average pore size of 1.4 μm [117]. The uniform TMP is around 0.5 bar. The volumetric reduction factor is 20, and in some huge equipment, the flow of the MF retentate is concentrated 10 times more in cascade by a second MF; this leads to a volumetric reduction factor of 200. Fluxes obtained industrially are in the order of 500 L/h m^2 during 10 h. The decimal reduction factor, defined as the ratio between the number of spores in the initial milk and the number of spores in the permeate, is higher than 4.

The Bactocatch plant is typically divided into two sections: the MF section and the high temperature treatment (HTT) section [131]. In the HTT section, the product is treated for a short time at high temperature (120 °C). For cheese application, the skim milk is separated into two fractions: the permeate with a low bacterial content and the retentate with a high bacterial content. The retentate is diverted to the HTT section and mixed with the cream needed for standardization. For extended shelf life products, the retentate is not re-mixed with the cream but is removed separately for further processing. As the retentate is considered a secondary product, higher concentration rates are often applied.

Like in all membrane processes (see "Membrane Processes"), membrane fouling occurs during skim milk MF [21, 103, 145]. Very rapid fouling is associated to the fact that most of the fat globules and some of the proteins are as large as or larger than the bacteria. Fouling mechanisms include complete pore blocking by bacteria and spores, partial pore blocking by bridging of casein micelles, adsorption to the membrane surface of serum proteins, and in-pore fouling by serum proteins [21, 103, 145]. To minimize the formation of the fouling layer, high cross-flow velocities (4–8 m/s) or backflushing techniques have to be used. In the Bactocatch system, high velocities are used with recirculation of the permeate to create a uniform TMP along the membrane. However, the use of high cross-flow velocities leads to a high-energy consumption and to problems with varying TMP along the membrane. [73] proposed a backflushing technique combined with reverse asymmetric membranes allowing the use of low velocities and resulting in lower energy costs. In the technique, the permeate is pressurized during a very short time interval (less than 1 s) and with a frequency around 0.2–1 s^{-1}. The use of a reverse asymmetric membrane allows the formation of a very open fouling layer mainly inside the porous structure. The technique, combined with reverse asymmetric membranes of pore size of 0.87 μm, allows the control of fouling, achieving very high (500 L/h m^2) and stable fluxes with 100% casein transmission and a high reduction of spores (decimal factor between 4 and 5) even at low velocities (0.5–1 m/s).

Separation of Casein Micelles from Soluble Proteins

Skimmed milk is a complex suspension of casein micelles (association of α_{s1}, α_{s2}, β, and κ-caseins with an average radius of 100 nm, 26 g/L) in an aqueous phase containing soluble proteins (mostly β-lactoglobulin and α-lactalbumin, with an average radius of ~ 5 nm, 7 g/L), minerals (mostly calcium and phosphate, 8 g/L), and lactose (50 g/L) [82]. The composition of skimmed milk may also change with processing conditions like change in ionic strength, which induces modification of

characteristics of casein micelles and protein and mineral composition [82].

Casein micelles are separated from soluble proteins by cross-flow MF (0.1 μm) in order to produce a retentate rich in casein micelles and a permeate rich in soluble proteins, minerals, and lactose [82]. Concentrated casein micelles can be recombined with cream for the production of cheese or use in production of dried native casein for food applications. In addition, permeate obtained from skimmed milk MF is a better starting material for the purification of individual proteins [21]. However, casein micelles may be retained at the membrane surface, which induces the formation of a fouling layer and a drop of permeability and selectivity [67]. Physicochemical conditions may have a strong impact on the overall performance of the process. For example, at high ionic strength, casein micelles may form a gel, which is compact and difficult to remove [82].

Hydrodynamic influences the performance of skimmed milk MF in terms of permeability. For example, the effect of turbulence on cross-flow MF of skim milk was shown to provide a significant increase in permeate flux [89]. The use of Kenics static mixer as turbulence promoter in the inorganic membranes (Membralox™) with pore sizes 50, 100, and 200 nm gave a flux increase of more than 500%, at *Re* number in the range 3000–10 000. Dynamic membrane systems are major techniques to limit membrane fouling and increase permeate flux. For example, vibratory shear–enhanced processes [6] and rotating membrane systems [61] were investigated successfully.

The separation of casein micelles from soluble proteins is a well-established technique. To our knowledge, there are few recent articles in this field. Recently, [28] showed that a submerged membrane vibrational systems had significantly better performance and separation efficiency than a conventional cross-flow MF system. Indeed, the submerged membrane system with membrane vibration increased the transmission of whey proteins (α-lactalbumin and β-lactoglobulin) while rejecting casein micelles. In addition, a low increase of TMP was observed during experiments conducted over 24 h indicating low membrane fouling. Ceramic membranes with pore size of 0.1 μm are commonly used for this application although polymeric spiral wound membranes can give similar performance when used at the same operating conditions (shear stress, TMP, and temperature) [21, 82]. A recent study [149] investigated successfully new stainless steel membranes that improve membrane resistance to fouling thanks to their well-defined pores shapes and surface properties.

Separation and Fractionation of Fat Globules from Whole Milk

Milk fat mainly consists of dispersed fat globules, with diameter between 0.1 and 15 μm, with an average size of 3.4 μm [71]. Their integrity is maintained by a thin membrane surrounding their internal core. The number of fat globules per milliliter is between 10^{10} and 10^{11}.

Fractionation by MF could be an alternative to homogenization, as MF is known for not damaging the fat globules membranes [71, 76]. [71] described the fractionation of milk fat globules using a 2-μm MF ceramic membrane. The permeate and retentate from several milk products, such as drinking milks, yoghurts, sour cream, camembert, Swiss cheese, and butters, were evaluated for their texture and organoleptic properties. Significant differences were found in the manufactured products. Except for butter, milks containing small fat globules found in the permeate lead to more unctuous products and finer textural characteristics versus products made with reference creams or with mainly large fat globules obtained in the retentates.

Separation of fat is generally realized by centrifugation. To avoid clumping of milk fat globules, the separation of fat globules usually takes place around 50 °C. Cream resulting from centrifugation contains about 40% fat. Advantages of membrane separation are a reduction in energy consumption and less damage to shear sensitive components, such as the milk fat globule membranes, when relatively low cross flow velocities are used [21]. This could result in enhanced stability of cream and improved sensory properties of consumer products.

Few works describe the separation of fat globules from whole milk. Recently, some authors have investigated the production of milk fat globule membranes known for their wide range of bioactivities, including anticancer, immunoregulatory, and stimulation of neural development [75, 83, 84]. Milk fat globule membranes can be added to supplemented infant formula to match the composition of human milk and their concentration can be adjusted to the children requirements. Flat globules are first separated by MF which can be associated to diafiltration (continuous addition of an aqueous phase). The milk fat globules membranes are then isolated using a specific procedure [75, 83, 84].

Concentration and Demineralization of Whey and Milk Ultrafiltration Permeate

Whey and milk UF permeate are nutritious protein sources, but their application in food or feed products requires their concentration and demineralization. Whey can be classified based on processing conditions: sweet whey (pH around 6), produced from rennet-coagulated casein or cheese, and acid whey ($pH \leq 5$) produced from mineral or lactic acid-coagulated casein [21, 23]. The concentrated serum proteins can be spray dried and used in food or feed applications. Depending on the protein content, these products are called whey protein concentrate (WPC, 35–80% protein) or whey protein isolate (WPI, 80–95% protein) [21].

Nowadays, whey and milk UF permeate are concentrated by evaporation, RO and/or NF [21, 139], Stoica et al. 2018). Compared with evaporation, membranes processes require less energy consumption and are easily scaled up. RO can be realized on the dairy farm for reducing transport costs and increase bulk tank capacity, and cheese can be produced from RO concentrated milk [125]. However, RO can be limited by the high pressure required and also by the relative low permeate fluxes obtained as well as by membrane fouling. As an alternative, [144] proposed forward osmosis to concentrate whey protein solutions. In forward osmosis, a concentrated draw solution and a diluted feed solution are separated by a semi-permeable membrane, where the osmotic pressure difference drives the water to flow from the feed solution to the draw solution. In contrast to RO, forward osmosis requires less pressure, which is also an advantage for treating viscous suspensions with high solid content.

NF is also used to demineralize and concentrate sweet whey streams prior to spray drying. NF was introduced in the 1990s as an alternative to reduce the amount of salts and to concentrate proteins in one step. An abundant literature is available in this field. For example, [134] give a detailed description of the NF process. NF reduces energy consumption but only leads to partial demineralization. NF membranes used in dairy applications have a high permeability for monovalent salts (NaCl, KCl) and a very low permeability for organic compounds (lactose, proteins, urea). More recent papers focused on the development of pretreatments that can improve ions rejection. For example, [105] realized the pretreatment of reconstituted sweet whey powder solution by anion-exchange resin to increase the molar ratio of Cl to Na and K. NF was then able to remove more than 90% of Na and K from the anion-exchange treated whey compared with less than 60% from the control whey. After being concentrated, whey is spray dried. However, high concentrations of lactate, either as lactic acid or dissociated into lactate ions, are shown to cause increased powder stickiness and so operational problems in the dryer [17, 29]. NF can be used to separate undissociated lactose from charged lactate anions in whey, to reduce the risk in the spray drying operation. [17] confirmed the advantages of these techniques at a semi-industrial scale.

Due to its high content in proteins and vitamins, whey can be used as an additive in baby food, cheese products, and candies. However, mineral salts affect its flavor and the functionality and value of the whey products. Desalination of whey is thus an important process and can be done also by ED or by ion exchange resins [21, 23]. In addition, ED with bipolar membrane (EDBM) is different from conventional ED since it uses the properties of bipolar membranes to split water molecules and may be also used to produce high purity bovine milk casein isolates. However, membrane fouling is one of the common problems in ED and this phenomenon could alter irreversibly the membrane integrity and could provide a decrease in the process performance. Also in EDBM configuration, a fouling identified as a mixture of $CaCO_3$ and $Ca(OH)_2$ was observed on both sides and inside the cationic membrane present in the stack. In addition, it was shown that conventional ED with pulsed electrical field can limit the protein fouling on the ion exchange membranes [23].

Recent works were proposed to solve the fouling limitations of the ED technique, reduce the duration of the process and/or improved removal of mineral salts. For example, [53] demonstrated the positive effect of pulsed electric field on lactic acid removal, demineralization, and decrease of membrane fouling during ED of acid whey. [33] confirmed that ED is a well-suited demineralization technology to remove lactate ions from acid whey. [98] investigated pulsed ED reversal, in which short pulses of reverse polarity are applied instead of a long pause period in ED with pulsed electrical field. In most ED commercial plants, the operating temperature for whey desalination is 10–20 °C, which implies a rather long operation. Increasing temperature makes the process faster and more efficient [99].

Fractionation of Whey Proteins

The fractionation of whey proteins is an increasingly important field in the food, nutraceutical, pharmaceutical, and biotechnology industries. A large literature is available on this topic with several recent reviews [4, 11, 43, 66, 102]. This ever-growing interest may be explained not only by the health benefits of these compounds and their physicochemical properties like gelation, thickening, and foaming in foods but also by the fact that whey was considered traditionally as a waste so the treatment and recovery of its valuable compounds can be considered as green technologies.

The two major whey proteins are β-lactoglobulin (35–65%) and α-lactalbumin (12–25%); minor components are immunoglobulins (8%), serum albumins (5%), and lactoferrin (1%) [102]. α-lactalbumin can be used in infant formula and as a nutritional additive, β-lactoglobulin has emulsifying and foaming properties, and immunoglobulins, serum albumins, and lactoferrin are both of interest in food and therapeutic applications [102, 150]. Several processes other than membrane processes are available to fractionate whey proteins, traditionally α-lactalbumin and β-lactoglobulin, the predominant whey proteins (> 60%). These include ion exchange chromatography, affinity chromatography, and precipitation [15]. The similar size (14 and 18 kDa) and pI (4.5–4.8 and 5.2) of these proteins (Table 1) complicate the separation process, resulting in inadequate yield and purity.

UF is an interesting alternative to chromatography for whey fractionation, which can be used at large scale. UF

can be very specific, for the purification of compounds that keep their properties. Using precipitation and UF, [68] reported a purity of 52–83% for α-lactalbumin and 85–94% for β-lactoglobulin, respectively. [88] compared different purification methods for β-lactoglobulin and obtained purities between 82.5 and 94.1%. To enhance the selectivity of the UF process, it is possible to make use of the specific properties of the proteins. Adjustment of pH and addition of salt influence the electrostatic and steric interactions between different proteins and between proteins and the membrane. For example, selectivities (defined as the ratio of the observed sieving coefficients of α-lactalbumin and β-lactoglobulin) of greater than 55 could be achieved at pH 5.5 and 50 mM ionic strength using a 30-kDa cellulose membrane [32]. However, with natural whey, decrease in both fluxes and selectivity was reported [54]. The difference in the flux and retention characteristics between model and natural suspensions is explained as a result of the interaction with other proteins resulting in aggregation in the natural whey. Membrane and operating conditions also affect the overall performance of UF fractionation. For example, [100] compared UF membranes of different hydrophobicities and different pore sizes in the enrichment of α-lactalbumin to the permeate from diluted whey solutions.

Several recent studies focused on the development of new processes to improve the separation of whey proteins from whey. For example, charged membranes were proposed to exploit the difference in isoelectric points of the proteins [13]. Cascaded UF (or multiple-stage UF), in which retentate and permeate from one membrane (stage) were transferred to the next or previous membrane (stage) as feed, were used to increase the yield and purity of the obtained components [107]. Also, ultrasounds were investigated successfully for the pretreatment of whey before UF [87] or associated to UF [2] with improved process performances. Lastly, enzymatic cross-linking with transglutaminase was tested for tuning protein size before UF showing an increase in whey protein recovery of 15–20% [146]].

Recovery of lactoferrin from whey has also been intensively investigated using membranes processes due to its multifunction and potential nutritional and therapeutic applications. The first reported studies used UF combined with appropriate pH conditions to exploit the physicochemical properties of these whey proteins (for example, [7, 104]). More recent articles proposed several alternative techniques, among them membrane techniques, to improve recovery and purity of the obtained lactoferrin. For example, membrane chromatography was shown to be very effective in improving separation efficiency and the technique has the advantage to be easily scaled up [129]. [133] demonstrated improved separation of lactoferrin and serum albumin using charged UF membranes. An ED technique was also developed recently to separate lactoferrin and immunoglobulins from a model whey solution [141, 142].

Wine

Wine is one of the most popular alcoholic drinks in the world, France, Italy, and Spain being some of the most important producers. Wines are obtained from the fermentation of grapes and their composition depends on the composition of the grape, grape ripeness at harvest, wine growing conditions, and wine-making practices. Their composition is also affected by yeast and bacterial strains and their metabolism. The average composition of wines (white and red) is shown in Table 2 [56].

Wine colloids include polyphenols, polysaccharides, and proteins [56]. In red wine, the most abundant phenolic compounds are anthocyanins and tannins; in white wine, hydroxycinnamic acids constitute one of the main groups of macromolecules found in wine that are polysaccharides. Polysaccharides may be divided into three groups depending on their origin: grape polysaccharides (pectin, pectin substances), yeast polysaccharides, and fungi polysaccharides like beta-glucan. Wine proteins are a mixture of grape proteins and minor extent proteins from autolyzed yeast. Red wines contain very few proteins, as they are precipitated by tannins. Moreover, white and rosé wines may have variable protein concentrations of up to a few hundred (10–500 mg/L), mainly originating from grapes.

In the overall production process, wine filtration is used to decrease turbidity and improve microbiological stabilization [56]. Turbidity is due to the presence of haze generated by macromolecules and particles in suspension. Turbidity can be measured with a turbidimeter and is expressed in nephelometric turbidity unit (NTU). Before treatment, the initial NTU can be as high as 2000 NTU [56]. After filtration, the turbidity of wines must be less than 2 NTU. Traditional

Table 2 Average composition of wines (adapted from [56])

	Concentration (g/L)	
Water	750–900	
Ethanol	69–121	
Glycerol	5–20	
Organic acids	3–20	
Minerals	0.6–2.5	
Nitrogen compounds	0.5–5	
(Amino acids and proteins)		
	White wine	red wine
Phenolic compounds	0.1–0.3	1.5–6
Polysaccharides	0.4–0.7	

filtration techniques include filtration on sheets and diatomaceous earth. Cross-flow MF is a possible alternative to increase wine limpidity and stability. It is becoming widely extended in the wine industry, due to advantages such as elimination of diatomaceous earth use, and its associated environmental and health restrictions, as well as the combination of clarification, stabilization, and sterile filtration in one single operation.

The consequence of fouling during cross-flow MF of wine is a decrease in permeation flux, affecting the economic viability of the process, and an increase in retention of some components, which may affect the organoleptic characteristics of the wine [56]. Fouling presents a very complex picture due to the complexity of wine composition, the influence of the membrane with wine colloids and particles, and operation conditions. Several studies have reported the negative effect of wine polysaccharides on the permeation flux. Membrane fouling is not directly related to its total polysaccharides content but rather to the composition, structure of these polysaccharides, and the balance between different groups of polysaccharides [137]. Proteins can also lead to very high and almost irreversible fouling. The related mechanisms are associated to hydrophobic interactions between the proteins and the membrane material as well as protein–protein interactions [132]. In addition, particle and yeasts can play a major role, whose impact depends on particle size. In addition, cake formation can even partially protect the membrane from internal pore blocking [132]). For example [19] investigated the respective impact of wine particles, i.e., *Saccharomyces cerevisiae* yeast and fines (lactic bacteria and colloidal aggregates), on the performances of cross-flow MF. It was found that yeast alone formed reversible deposits, in relation with their hydrophilic character and uncharged properties. By contrast, fines formed a coherent and adherent cake above a given TMP.

The nature of the membrane has a major influence on fouling during cross-flow MF of wine. The interactions of wine components with the membrane surface play indeed a major role in membrane fouling. For example, [132] from MF clarification of a white wine showed that polypropylene membranes yield significantly higher fluxes than polyarylsulfone membranes, both having the same pore size (0.2 µm). Using a model solution for wine, they showed that polyphenols and polysaccharides were very little adsorbed by polypropylene but strongly adsorbed by polyethersulfone membranes. In addition, it was suggested that aggregates of polyphenols and polysaccharides present in red wine had a strong contribution to adsorptive fouling and that the interaction between polyphenols and the membrane surface was the main phenomena.

The better understanding of fouling phenomena during cross-flow MF of wines combined with the introduction of backpulsing has led to various commercial plants [56]. For example, the Oenoflow XL-A system (Pall) is designed to meet the requirements of large wineries. Clarification is achieved in a single process step without the need for filter aids or centrifugation, and MF does not show any significant impact on the organoleptic characteristics of the wine. The system utilizes poly(vinylidene fluoride) hollow fiber membranes which allow exposure to aggressive cleaning solutions. In addition, the Oenoflow XL-A system is fully automated and incorporates backflushing. The Flavy FX system (Bücher Vaslin) uses hydrophilic polyethersulfone membranes specifically adapted to wine filtration. The asymmetrical structure of the membrane contributes to reduce fouling, and its hydrophilic characteristic has an efficient role for controlling the adsorption of polyphenols and polysaccharides. In addition, the system incorporates backflushing for regular unclogging of the membranes. Other systems have been tested at lab scale to reduce membrane fouling during wine clarification. For example, [57] investigated a rotating and vibrating filtration module with a complex hydrodynamic generated by a 3 flat blade impeller in a confined cell. With a hydrophilic polyethersulfone membrane, a permeability increase of 300% was obtained.

In the wine industry, some recent trends can be underlined. Wines with reduced alcohol content are increasingly requested by consumers. To obtain wines with reduced alcohol content, membrane processes including NF, RO, and membrane contactors can be used as alternative to traditional techniques like evaporation [58]. To reduce the alcohol content in wine, two methods are possible: either the reduction of sugar concentration of musts or the dealcoholization of wines [58]. Sugar control in beverages such as grape must can be obtained by membrane processes like NF. To reduce the loss of volatile aroma, during NF, several processes are sequentially implemented. For example [119] evaluated the combination of pervaporation and NF to obtain a full flavored low alcohol white wine. The two-stage NF process was used for sugar reduction of must; pervaporation was realized for recovery of aroma precursors from grape before the two-stage NF and restitution of the flavor precursors. New products are also requested by consumers. For example, [147] described the preparation of mulberry wines which has several pharmacological effects. MF was proved to be an efficient technique for sterilization and clarification to produce a wine of high quality and stability. Some membrane processes are also emerging like ED used in reagent-free pH correction and tartrate stabilization of wines which is a main source of wine instability by precipitation of the tartaric salts [58]. Compared with other methods like cold stabilization and UF, ED limits the loss of wine and of flavored components. However, membrane fouling by polyphenols and polysaccharides is a major limitation as it reduces electroconvection [122].

Beer

In the food industry, the brewing industry holds an important position [63]. Beer is the fifth most consumed beverage in the world behind tea, milk, and coffee. Beer clarification is one of the most important operations in the brewing industry [135]. It consists of rough beer filtration in order to eliminate yeast and colloidal particles responsible for haze, with the aim to ensure the biological stability of the beer. Fermentation of beer is done by yeast cells, converting starch-derived maltose into alcohol and CO_2. Fermented beer is therefore a complex mixture of fluids, cells, aggregates, and macromolecules. Turbidity is due to the presence of yeast cells and haze particles, which are aggregates of proteins and polyphenols. The conventional dead-end filtration with filter aids, such as diatomaceous earth (Kieselguhr), is the usual industrial technique for beer clarification. However, due to economic, environmental and technical limits, cross-flow MF has been developed and applied in several industrial plants.

During beer clarification by MF, severe membrane fouling is observed which leads to low permeate fluxes (typically below 100 L/h m^2), protein and aroma compound retention, and extensive cleaning procedures [41]. Membrane fouling can be caused by pore size constriction, pore blocking or the deposition of cells, cell debris, and/or other particles, such as macromolecules or macromolecular aggregates, on the top surface of the membrane. In addition, macromolecules present in beer (proteins, polyphenols, polysaccharides, etc.) can cause significant fouling, even though they are much smaller than the pore size of MF membranes.

The membrane pore size is an important parameter to control flux and filtrate quality during beer clarification. The optimized nominal pore diameter is 0.45 µm to obtain a balance between flux and selectivity in comparison with the 0.2 and 1.3 µm pore size [65]. Ceramic membranes are preferred to polymeric membranes as they can withstand harsh cleaning conditions. However, the obtained fluxes are usually much lower. Enhanced surface hydrodynamics have limited effect on improving flux and retention as the surface flow conditions have little influence over the dominant in-pore fouling and on the transmission of macromolecular solutes. However, high-frequency backflushing and reversed membrane morphology were shown to lead to high flux enhancement [65]. The reversed membrane pore morphology is obtained by changing the configuration and feeding the fluid to the substructure side of the membrane and taking out the permeate from the usual retentate side. The TMP is then applied in a reversed mode.

Several commercial MF plants are used for beer clarification [135]. The rough beer is first cooled down to 0 °C to induce chill haze, which is also retained by the membrane and will not cause turbidity in the bottled beer. Commercial systems mostly use hydrophilic, polyethersulfone membranes, with pore diameters in the range of 0.45–0.65 µm, in the configuration of hollow fiber or flat sheet modules. Some commercial systems use a centrifuge separation step prior to MF, which eliminates the yeast cells and large aggregates. However, the costs of the centrifuge step are quite large compared with the ones of the MF step; therefore, most industrial plants do not involve a centrifugation step.

Tank bottom recovery constitutes also an important application of membrane processes in the brewing industry, and several industrial units are used with ceramic 0.4–0.8 µm or polysulfone 0.6 µm MF membranes [63]. In order to recover beer from tank bottoms, natural sedimentation, centrifugation, or filter press can be used. As an alternative, MF can produce a high-quality beer including flavor and haze, with minimal loss of color and bitterness.

More recent trends include the development of low-alcohol and alcohol-free beers. Non-alcoholic beers are usually defined by an ethanol concentration lower than 0.5%vol. Dealcoholization of beers is realized using biological processes where fermentation is controlled to limit ethanol production, the use of specific yeast strains that consume or do not produce alcohol, thermal ethanol removal, or ethanol removal by membrane processes [101]. Membrane processes for dealcoholization of beers have the main advantage of operating at mild temperatures compared with traditional thermal technologies. These membrane techniques include dialysis, RO, and pervaporation. However, several drawbacks are associated to these technologies: low flux for dialysis, fouling and high-energy consumption for RO, and loss of volatile compounds for pervaporation. Other processes are proposed like non-porous membrane distillation [112] and forward osmosis diafiltration [9]. In forward osmosis, the osmotic pressure difference between the two sides of a semipermeable membrane drives the solution with a lower fouling than in RO. Water and ethanol are simultaneously removed; diafiltration is used to add water, reducing the alcohol content.

Another recent trend in the brewing industry is the use of enzymes [37, 38]. Enzymes are used to digest cell walls, release amino acids, and breakdown starch into fermentable sugars. Such multi-enzyme blends are commercialized under the trade names of Ondea® Pro and Brewers Clarex. These multi-enzyme blends can be used to improve the brewing process, produce a gluten free beer, or use raw barley or a new raw material to create new flavors. Several recent studies demonstrated the successful use of combined enzymatic and cross-flow MF for clarification and stabilization of beer. For example, [37] tested the commercial Ondea® Pro

preparation and [38] tested the commercial enzyme preparation of fungal origin Brewers Clarex.

The treatment of wastewater is also of increasing concern in the brewing industry. Wastewater is the main source of waste, and it is estimated that between 3 and 10 L of waste effluent is obtained per liter of beer [106]. The components of the wastewater include sugars, soluble starch, ethanol, and suspended solids. Several techniques are able to treat wastewater from the brewing industry like physical, chemical, and biological treatments and microbial fuel technology. Membrane processes are also possible alternatives using membrane bioreactors [33, 34] or MF [49]. For example, Chen et al. [33, 34] investigated the treatment of brewery wastewater using an advanced anaerobic membrane reactor. The anaerobic membrane bioreactor integrates anaerobic digestion and membrane filtration, where the membrane completely retains all suspended solids. Dizge et al. [49] realized the treatment of brewery wastewater using electrocoagulation, ultrasonication, and sono-electrocoagulation associated to cross-flow MF.

Fruit Juices

Fruit juice production is one of the most important sectors of the fruit juice industry. During the last years, the consumption of fruit juices has significantly increased due to perception of juices as a healthy natural source of nutrients [18, 24, 39]. Fruit juices contain low molecular weight solutes such as sugars, organic acids, vitamins, pigments, and high molecular weight solutes such as proteins, polysaccharides (pectin, cellulose, hemicelluloses, lignin, and starch), and microorganisms. During the last decades, membrane processes have been increasingly used in fruit juice industry. These techniques can produce fruit juices with high quality, thanks to their high selectivity and low operating temperatures. They are mainly used not only for concentration and clarification but also for isolation and purification of specific compounds (such as aroma and bioactive compounds).

Fruit Juice Clarification

Some fruit juices present natural turbidity due the presence of insoluble matter such as pectin, starch, and cells from the juice [50]. Depending on the application of the fruit juice, a clarification process is required. For example, clarified fruit juices are needed for the production of clear beverages (soft drinks, natural aromatic waters, alcoholic beverages, cold teas, etc.), candies (melting products), and pastries (natural essences, translucent fruit sauce). Conventional clarification usually involves sequential batch processes, such as enzymatic pretreatment, clarification with bentonite, gelatin or diatomaceous earth, and pasteurization. Introduced in the late 1970s, MF and UF are alternatives to traditional clarification techniques. Advantages of MF and UF are increased juice yield, possibility of operating in a single step, reduction in enzyme utilization and working times, elimination of needs for pasteurization, no requirement of filter aids, and production with a continuous process [24, 115].

Concentration polarization and fouling phenomena represent important limitations in the performance of fruit juice clarification using UF or MF. Membrane characteristics, juice properties, and operating conditions, such as recirculating flow, TMP, and temperature, are important factors influencing membrane fouling and, consequently, the process performance. The influence of operating conditions on membrane fouling in cross-flow MF and UF of different fruit juices has been widely investigated (for example, [27, 47, 91]). Like in other UF and MF applications, the permeate flux shows a linear dependence with pressure lower than a critical TMP while at higher pressures, the permeate flux approaches a limiting value. Higher flow rates enhance the wall shear stress on the membrane surface reducing the concentration polarization layer associated to an increase in permeation flux. Permeate flux is also increased by using higher juice temperature due to a reduction of juice viscosity and an increase of diffusion coefficients of macromolecules [27].

In general, the permeate flux increases with membrane pore size (in case of MF) or MWCO (in case of UF) while the retention of sugars, pigments, and potential haze precursors (phenolics and proteins) decreases as membrane pore size (or MWCO) increases [70]. The selection of MF or UF membranes for fruit juice clarification must consider several parameters including the viscosity of the raw juice, the retention of specific compounds, the membrane performance, and the membrane cost [24]. Juices with high solids content and viscosity require the use of tubular modules or plate and frame modules with large spacers. Particle clogging can occur in the narrow flow path of hollow fiber or spiral wound membrane modules reducing permeate flux and making membrane cleaning more difficult. Consequently, the use of thin channel devices requires additional pretreatments of the fruit juice to reduce solids content and viscosity.

Commercially available membranes used in fruit juice clarification are usually organic polymers such as polysulfone, polyethersulfone, polypropylene, polyamide, cellulose acetate, polytetrafluoroethylene, and poly(vinylidene fluoride) ([18, 24, 39]). Ceramic membranes are also used for their higher chemical resistance and longer life. The most used devices are tubular membranes (with an inner diameter of 5–10 mm), capillary membranes (inner diameter of 1–1.5 mm) and plate-and-frame membrane modules. The tubular configuration is associated with low packing density and high membrane replacement costs. Vibrating membrane systems able to develop high shear rate at the membrane surface may also be used for juice

with high solids content and viscosity. An immersed membranes configuration has also been tested for concentration of delicate fruit juices that could be damaged by the high cross-flow velocities (1–7 m/s^{-1}) applied in classical membrane configuration like ceramic tubular membranes [115].

Pretreatment plays an important role in fruit juice clarification by MF or UF. Low permeate fluxes can be obtained during juice filtration due to its relative high content of pectin, lignin, and hemicellulose. Enzymatic treatment and juice centrifugation are common pretreatment methods. [50] investigated the influence of three pretreatments before passion fruit juice MF: centrifugation, enzymatic liquefaction, and chitosan coagulation. The enzymatic pretreatment reduced the juice viscosity, and the centrifugation step decreased color and turbidity. Chitosan coagulation was the most promising pretreatment, since it gave the highest decrease of color and turbidity and the highest permeate flux. It was also observed that the predominant membrane fouling mechanism depends on the pretreatment. Fouling occurs mainly by cake formation for fruit juice pretreated by centrifugation or enzyme and by internal pore blocking for fruit juice pretreated by chitosan.

Recent interest in eco-responsible techniques (less energy required and less transport of the products obtained) has also been investigated in the field of fruit juices. [110] recently proposed the concentration of citrus fruit juices using traditional solar drying setups and membrane pouches that are easier to handle and transport to local urban markets. The membrane pouches were made from a polymer film cut into two pieces and then joined together on three sides with a heat sealing machine. The technique is suitable for small-scale producers in rural areas of tropical countries like rural Mozambique. The results obtained at realistic weather conditions can help small-scale producers to use the technique.

Fruit Juice Concentration

Fruit juices are usually concentrated to allow easier and cheaper storage, transportation, and distribution, as well as better conservation. The classical methods of juice concentration, such as thermal evaporation, usually employ high temperatures to remove water. However, heat can cause undesirable changes in the product sensory and nutritional properties, such as color and flavor changes, and reduction in the nutritional value. Membrane concentration processes are alternatives to thermal evaporation. They are able to concentrate juices at room temperature, causing little or no damage to the product [10, 81]. Advances and developments of membrane technology in this field have been reviewed by [81].

Reverse Osmosis

Fruit juice concentration by RO has been used in the fruit processing industry for about 40 years [18, 24]. RO is based on the use of dense membranes operating at a TMP higher than the osmotic pressure of the solution in order to allow the permeation of water from the side at high solute concentration to the low one. Typically, high retention of nutritional, aroma, and flavor compounds can be achieved with the attainment of high-quality fruit juices.

The advantages of RO over thermal evaporation are low damage to the fruit juice, reduction in energy consumption, and lower capital investments, as the process is carried out at low temperatures. In addition, it does not involve phase change for water removal. RO concentration has been investigated for a large range of fruit juices, including apple, citrus, grapefruit, and others like juices from kiwi, pineapple, watermelon, passion fruit, and tomato [18, 24]. Several studies have demonstrated the effect of the membrane and operating conditions, on the permeate flux and retention of juice constituents, especially aroma compounds and sugars. Increased pressures can result in higher flux, reduced process time, and higher rejections of aroma compounds. At higher temperature, both permeate fluxes and permeation rates of volatiles are increased [36]. Different membrane configurations may also affect the retention of flavor compounds during fruit juice concentration, like spiral wound system and plate-and-frame configuration.

Some macromolecules present in fruit juices, especially polysaccharides, tend to form a gel layer at the membrane surface. In order to minimize this problem, enzymatic hydrolysis has been proposed to break down the methoxylated pectin, reducing the juice viscosity and, thus, favoring mass transfer across the membrane [5]. RO membranes have a major drawback in spite of their high selectivity and solute retention capacity. The high osmotic pressure of fruit juice limits its degree of concentration to the required level. Different studies have shown that the final concentration of fruit juices is lower than about 25–30°Brix with the most efficient flux and solute recovery [18, 24]. Therefore, RO can be used as a preconcentration step prior to other processes like freeze concentration or evaporation, to reduce energy consumption and increase production capacity. Multi-stage RO is also a possible alternative that is used industrially. For example, [8] investigated an industrial full-scale plant based spiral wound membrane modules to concentrate apple juice and identify the optimal conditions to obtain a specified apple juice product of high concentration.

MD

In MD, the water flux goes from the warm side to the cold side due to the temperature gradient between two aqueous

solutions separated by a macroporous hydrophobic membrane. The process is performed at atmospheric pressure and at temperature that may be much lower than the boiling point of the solutions. The driving force is the vapor pressure difference between the two solution–membrane interfaces due to the temperature gradient. Therefore, MD can be used to concentrate solutes sensitive to high temperature. The technique has received a great attention for concentrating fruit juices [81].

Different configurations have been used depending on the mechanism to obtain the required driving force. In direct contact membrane distillation (DCMD), water at lower temperature than the juice is used as a condensing fluid in the permeate side of the membrane. This configuration offers several advantages due to the possibility to operate at relative low temperatures and to achieve high-quality concentrates with high content of soluble solids (60–70°Brix). Several juices have been treated using DCMD like orange [22] and apple juices [74]. Several parameters affect the process performance, in terms of permeate flux and retention of fruit juice components, such as membrane type, feed juice concentration, operating temperature, flow rate, and UF pretreatment [81]. In vacuum membrane distillation (VMD), the membrane separates the liquid feed (the juice) from a downstream gaseous phase kept under vacuum. This process has been used for example for the recovery of volatile aroma compounds from pear juices [48]. These last years, an increasing interest has been paid to osmotic membrane distillation [14].

OD

OD is a membrane process, also known as osmotic evaporation, membrane evaporation, or isothermal membrane distillation. As compared with RO and MD process, the OD process has potential advantages which might overcome the drawbacks of RO and MD for concentrating fruit juice. Indeed, RO is limited by the osmotic pressure, and during MD, some loss of volatile components and heat degradation may occur due to the heat requirement for the feed stream in order to maintain the water vapor pressure gradient.

In OD, the membrane separates the dilute solution (the juice to be concentrated) from a salt solution (hypertonic solution) preventing penetration of the pores by aqueous solutions due to its hydrophobic nature. The difference in solute concentration generates a vapor pressure gradient across the membrane causing a vapor transfer across the pores from the dilute solution to the concentrated one. The process can be carried out at room temperature and low pressures (typically lower than 140 kPa) so avoiding thermal and mechanical damage of juice components. Disadvantages of OD for fruit juices concentration include a low evaporative capacity (3 L/m^2 h) with a long processing time and the necessity of an inactivation enzyme pretreatment [81]. Other drawbacks are related to economic aspects, such as production costs higher than the thermal evaporation and high cost of membrane replacement.

The removal of water by OD has been investigated for a variety of fruit juices including pineapple, camu-camu, passion fruit, kiwi, orange, and apple juices [24, 25]. Recent fruit juices treated by OD include Nagpur mandarin fruit juice [90] and cactus pear juice [130]. The water flux depends on different parameters such as flow rate, temperature, and concentration of both feed and salt solutions.

Salt solutions, such as $CaCl_2$, $MgSO_4$, K_2HPO_4, and NaCl, are typically used as stripping solution at the downstream side of the OD membranes [24]. For OD applications, membranes made of hydrophobic polymers such as polyethylene, polypropylene, polytetrafluoroethylene, and polyvinylidene may be used. For example, Liqui-Cel Extra-Flow membrane contactors with a shell-and-tube configuration designed for both laboratory and industrial applications are commercialized by Membrana (Charlotte, USA). These modules contain microporous PP hollow fibers with external diameter of 300 μm and a mean pore diameter of 0.2 μm. The total membrane surface area ranges from 1.4 to 135 m^2 [1].

Integrated Membrane Processes

Concentrating fruit juice by integrated membrane processes is attractive, particularly for industrial production [24]. Fruit juices such as orange juice have high solids and pectin content and are therefore viscous, which results in low permeate flux through RO or OD membranes. Also, using a single-stage RO system, concentrations cannot reach higher values than 25–30°Brix due to osmotic pressure limitation. By using MF or UF as a pretreatment for separating the suspended solids and pectins from juices, the viscosity decreases and the permeate flux through RO or OD increases. Therefore, high-quality product and lower energy consumption can be achieved with integrated membrane processes. Figure 3 shows an example of an integrated membrane process for concentration of clarified kiwifruit juice by OD with a UF pretreatment [25]. Another example is an integrated UF/RO/OD process based on a first step of UF for clarification of the centrifugated Nagpur mandarin juice, followed by concentration steps by RO and then OD [90].

Deacidification of Fruit Juice by Electrodialysis

In the beverage industry, ED is an interesting method for deacidification of fruit juice compared with salt precipitation, which involves addition of chemicals, and to ion-exchange resins, which only slightly modify the sensorial characteristics of fruit juices [136]. ED has shown

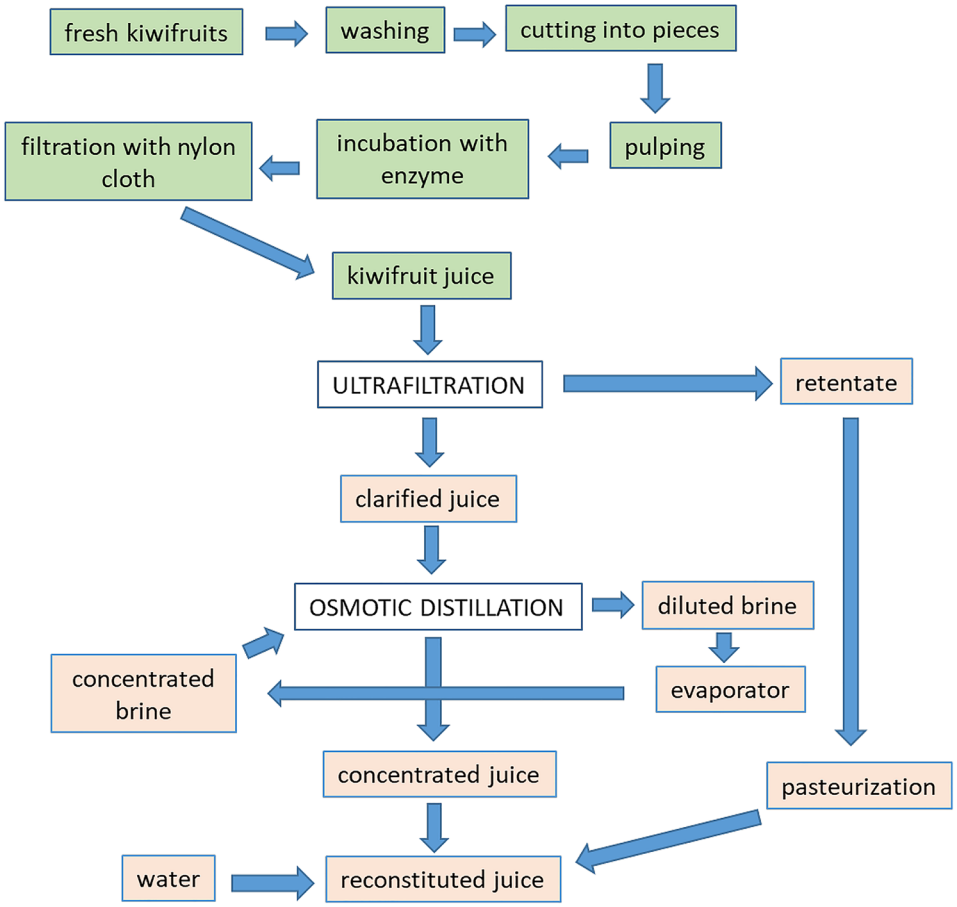

Fig. 3 Integrated membrane process for the production of highly nutritional from kiwifruit juice. Adapted from [25]

satisfactory performances for deacidification of various fruit juices such as orange, grape, pineapple, castilla mulberry, naranjilla, and araza. ED with bipolar membranes (EDBM), for the deacidification, has additional advantages, since the electrohydrolysis of water produced in the bipolar membrane allows producing H^+ and OH^- ions, which can be used for the deacidification, and the production of organic acids, that can be used to compensate the higher energy cost compared with other processes. Membrane fouling is the main limiting factor of the process, but it can be reduced by modifying current density and flow rate specifically for each juice [136]. The best ED performances are obtained with clarified and centrifuged fruit juices.

Recent results led to identical conclusions. For example, EDBM was shown to be slightly less damaging than salt precipitation during deacidification of cranberry juice production, and was up to around 20% more eco-efficient than salt precipitation [62]. The application of a pulsed electric field (an electric pulse is applied and stopped for a given time) during EDBM on cranberry juice deacidification can reduce fouling and increase current efficiency [109].

New Products

The development of new food products and the recovery of bioactive compounds have been proposed these last decades. Some recent examples are given below. [138] produced a goat whey orange juice beverage clarified and concentrated by MF. The addition of whey proteins into orange juices benefits from the bioactive activities of the whey proteins like antioxidant and inhibition of lipid oxidation. Healthy food products are also of major concern. In that sense, low-sugar fruit juices can be produced using membrane techniques. For example, [120] produced a low-sugar apple–cranberry juice based on an UF/diafiltration technique. The percentage of reference intake of sugars associated with the consumption of 250 mL juice was reduced from 27.0 to 20.7 and 15.7%, after UF and diafiltration, respectively.

The recovery of bioactive compounds from fruits has been also increasingly reported [102]. Polyphenols are known for their health benefit effects related to the reduction of heart disease, neurodegenerative disorder, and cancer [40]. Membrane techniques are interesting alternatives for extraction and purification. For example, NF was shown to be an effective method for concentration of phenolic

compounds from strawberry juice without color degradation [11]. NF was also used successfully for concentrating polyphenolic compounds from grape seed extracts without loss of polyphenolic compounds [92]. Natural aroma is also an important compound in food industries. They were obtained from fruit juice hydrolates by pervaporation, from plum, apple, blackcurrant, or cherry [45]. Pervaporation can significantly improve product quality and lengthen shelf life as demonstrated at laboratory scale and semi-industrial setup.

Sugar

Membrane processes have been proposed in the sugar industry to replace or to be added to traditional major operations [93]. These applications concern both the beet and cane sugar industries. In the beet sugar industry, applications reported concern pulp recycling, raw juice purification, demineralization of beet juice, and preconcentration of thin juice. In the sugarcane industry, they include raw sugar cane juice purification, concentration of clarified cane juice, molasses treatment, and decolorization of remelted raw sugar. A recent and increasing attention is also paid to the treatment of sugar vinasse [121].

Purification of Sugarcane Juice by UF

The production of sucrose from sugarcane juice includes several downstream unit operations [3]. The classical purification process (double-sulfitation) involves calcium hydroxide (lime) and sulfur dioxide addition to clarify the raw juice stemming from a sugar cane mill. In result, the suspended colloids of the raw juice coagulate and precipitate along with calcium sulfite [3, 93]. The clarified juice is then concentrated by evaporation and bleached by a second sulfitation. Finally, the product stream is crystallized to obtain white sugar crystals. However, the double-sulfitation process suffers from inefficient removal of substances from the raw juice, like gum, ashes, silica, colorants, and reversible colloids, and an inefficient removal of sulfite from the product stream. Therefore, MF and UF have been investigated to replace the second sulfitation step for the purification of clarified sugarcane juice (Fig. 4). The UF of clarified sugarcane juice can be done using spiral wound or flat sheet polymeric membranes or ceramic tubular filtration systems [80]. The filtrate obtained had an increase of 1.5–3 unit of juice purity, compared with the increase of 0.5–1 unit obtained using the liming-sulfitation process. In addition, membrane clarification yielded improvement in juice clarity with nearly 60% reduction in color as well as reduction in the inorganic contents of the juice.

The clarification of sugarcane juice by UF and MF has been extensively studied both in laboratory and factory trials. For example, [69] conducted a pilot test on the UF of sugarcane juice in a white sugar factory in northern India. The sugarcane juice was clarified at 91–97 °C using polymeric spiral wound membrane modules in a pilot plant with a production of 10 m^3/h. The membrane modules were found resistant to elevated temperatures and showed satisfactory separation with an average purity rise of 0.9 units, 31% lower turbidity, and 47% lower color in the permeate. However, the average flux was low (7 L/m^2 h). Like in other food membrane processes, membrane fouling is a significant limitation in the UF of clarified sugarcane juice [118]. Sugarcane juice contains 3–5% of soluble solids in the form of colorants, color precursors, etc., and 0.85–1.45% of soluble solids in the form of organic non-sugars such as proteins, polysaccharides, and waxes. The polysaccharide component in sugarcane juice is an established membrane foulant. In addition, the presence of divalent cations, e.g., Ca^{2+}, increases the gel formation by polysaccharides. Recently, [124] underlined the importance of pretreatment of factory sugarcane juice before UF to increase permeate flux and juice quality and reduce membrane fouling. Flocculation before sugarcane juice clarification minimizes the impurity loading and thus membrane fouling. However, the current flocculants tested in the sugar industry are still under investigation [124]. Integrated processes are also interesting options for purification of sugarcane juice. [96] demonstrated that integrated membrane processes were an interesting alternative to obtain both high color removal and satisfactory permeate flux in refining sugarcane juice. The integrated membrane process consisted of tubular loose UF, spiral wound tight UF, and finally spiral-wound NF.

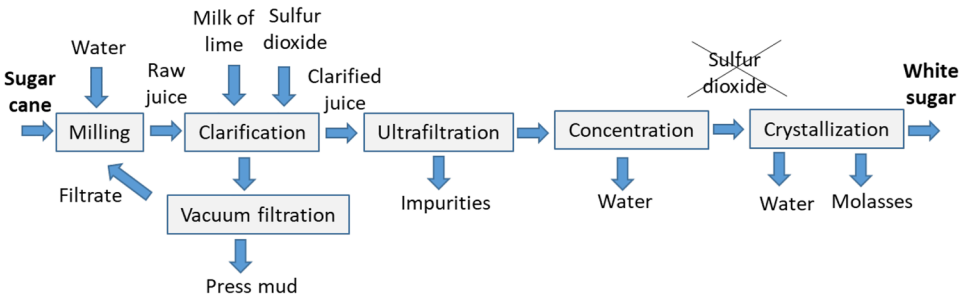

Fig. 4 Application of UF in cane sugar refining. The aim is to eliminate the second sulfitation step of clarified and concentrated sugar cane juice. Adapted from [3]

Demineralization of Sugar Juice

The sugar juice obtained after purification contains alkali cations (Na^+, K^+, Ca^{2+}) that must be removed [93]. Several processes can be used, i.e., ion-exchange resins, coagulants, adsorbents, and ED. The implementation of ED in the European sugar industry dates back to the 1990s. ED is an alternative or additional step to the classical demineralization of the sugar juice by ion exchange. ED is reported to offer a decrease in waste effluents, pollution load, volume of molasses, and capital cost in a continuous operation.

Some studies have been conducted on both laboratory and pilot scales (for example, [, 59, 64]). Elmidaoui et al. [59] used a specific membrane, Neosepta AXE 01 (Tokuyama, Japan). The membrane was used in an ED pilot plant of 2.5 m^2 at a production rate of 24 m^3/day. The juice quality was improved by demineralization reaching 9% in purity and 10% decoloration rate. At the end of the ED, 75% of Na^+, 86% of K^+, and 65% of Ca^+ were removed and no fouling was observed during 200 h. ED was then applied to remove melassigenic ions for Moroccan beet sugar industries [60]. Three solutions (juice, syrup, and mother liquor) were successfully treated by ED with improved quality and purity.

Processing of Sugarcane Vinasse

Vinasse is the aqueous waste generated by the distillation of fermented sugarcane juice or molasses during ethanol production [77]. Vinasse is an acidic stream (pH 3.5–5) with a high organic load. It contains nitrogen, phosphorus, and potassium. The production of vinasse is rising with the increasing demand in ethanol; therefore, its treatment has become a major issue. The literature on sugarcane vinasse treatment using membrane processes is abundant [77]. Two major membrane processes have been proposed. MF can be used, however, due to the high organic load of vinasse; pretreatments are required such as coagulation/flocculation [97]. The major drawback of MF processing is the high fouling of the membranes. Anaerobic membrane bioreactors are also possible alternatives. Again, membrane fouling is a major limitation reducing the permeate flux and increasing the processing time. These negative effects may be reduced by making use of the numerous advances in the membrane bioreactors field, especially in waste water treatment. For example, [121] demonstrated the potential of an experimental plant consisting of an upflow acidogenic reactor and a continuous stirred methanogenic reactor, fitted with submersed MF hollow fiber membranes.

Other Applications

Other applications of membrane processes are currently reported in the food industry including ED for separation, concentration, and purification of organic acids from fermentation broth and membrane emulsification in integrated processes for innovative food.

Electrodialysis of Organic Acids from Fermentation Broth

Organic acids, for example, lactic, succinic, gluconic, or citric acid, are widely used in the food industry, as acidulants, nutrients, and preserves [94]. Production at the industrial scale is mainly achieved by fermentation from molasses, starch hydrolysates or sugars. Several unit operations are usually required including one or several precipitation stages, which produce large amounts of effluents with a high salt content. ED is also a possible technique for separation, concentration, and purification of organic acids from fermentation broth by reducing the amount of effluents and involving less chemicals. Some recent papers underline the key role of organic acids in several industries especially in the food industry (for example, [46, 95]).

In EDBM, the bipolar membrane, consisting of a cation-selective layer and an anion-selective layer, is used to separate the system into functional chambers [94]. With the capability of bipolar membranes to dissociate water into H^+ and OH^-, the organic salts can be converted into organic acids. With a suitable applied voltage (e.g., 0.9–1.1 V), the water dissociation rate can be 50 million times faster than that in aqueous solutions. Moreover, the process requires much less energy than an electrolytical production process. Several organic acids have been produced using EDBM, including acetate acid, propionic acid, citrate acid, and gluconic acid.

For example, lactic acid is produced by fermentation followed by a two-stage ED recovery process (Fig. 5) [20]. The major application of lactic acid in the food industry is as an additive and preservative. In order to increase the fermentation yield, the pH is adjusted by addition of a base. The resulting fermentation broth contains the lactic acid as calcium, ammonium, or sodium salt and several organic and inorganic fermentation residues. The largest impurities, such as bacterial cells and high molecular weight residues, may be eliminated in a first clarification by MF. The suspension is then concentrated by conventional ED to improve the removal of non-migrating species like remaining sugars. After conventional ED, the acid salt is then converted into its free acid form by EDBM. Improving the overall efficiency of a two-stage ED recovery process

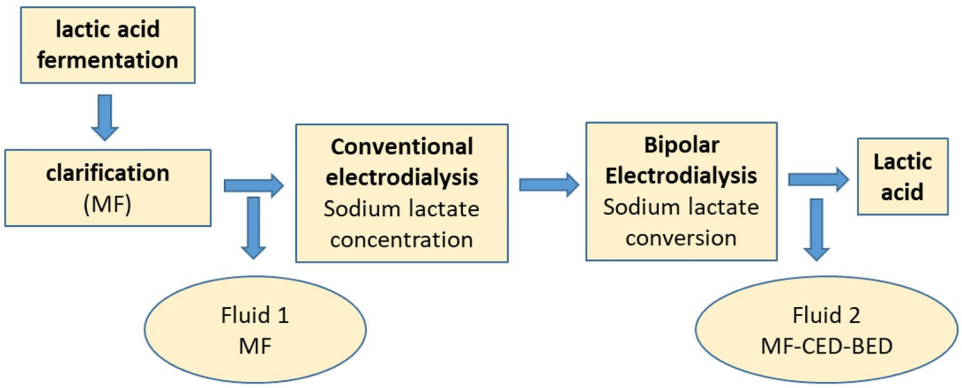

Fig. 5 Schematic diagram of a classical two-stage ED recovery process for lactic acid production. BED: bipolar electrodialysis. Adapted from [20]

generally involves adding extra purification operations before and/or after EDBM, such as NF. For example, NF can be used for clarification of the fermentation broth as well as a final purification step after lactate conversion [20]. More recently, [143] proposed the integrated operation of fermentation and EDBM under continuous operation. Both continuous fermentation and continuous EDBM were investigated separately before being integrated in the same setup.

Membrane Emulsification

Membrane emulsification has been introduced some 30 years ago as an alternative to other methods of emulsification [31, 140]. Emulsions are prepared by injecting the dispersed phase through the side of a porous membrane, and the droplets formed are detached by the continuous phase flowing on the other side of the membrane. Typically, the technique can be realized by injecting through the membrane the dispersed phase (direct membrane emulsification) or a coarse emulsion (premix membrane emulsification). Premix emulsification can include several passes through the membrane to decrease the average droplet size and the droplet size distribution. A very large range of emulsions and particles have been prepared using membrane techniques including simple emulsions, multiple emulsions, and colloidal dispersions such as polymeric, lipid nano- and microparticles, nano- and microcapsules, and microbubbles [140]. The advantages of membrane emulsification over other emulsification techniques like high pressure homogenization are the low shear stress involved, keeping the properties of shear sensitive ingredients, the ease of scaling up, and the lower energy consumption [31, 140].

In the last two decades, membrane emulsification has become a well-established technology [31, 140]. Several membrane emulsification devices are commercialized, for example, by SPG Technology (Japan), Emulsar (France), Micropore Technologies (Great-Britain), and Kinematica (Switzerland). It is now realist to implement at large-scale membrane emulsification setups in industries. The setups can include the presence of membrane rotation, vibration, or oscillation, so droplets can detach easily; also, the high shear stress created in this way at the membrane surface helps to obtain droplets with small size (typically lower than 0.1 μm). Another trend is to use microsieve membranes that are particularly well-suited for these applications thanks to their well-defined pore shapes that control the droplet size and also that can reduce somewhat membrane fouling [140].

In the food industry, membrane emulsification has several potential applications for the preparation of beverage, dairy products, and others [31]. The first papers published in the 1990s were particularly interesting and described industrial applications in the dairy industries. Butter, margarines, and fat-based spreads are well-known dairy emulsions [42]. Other dairy emulsions include yogurts, processed cheeses, and other systems containing emulsions droplets. Membrane emulsification can be a suitable alternative to other emulsification processes and several studies were done on this topic. For example, [123] prepared oil/water emulsions using Shirasu porous glass (SPG) membranes. The dispersed phase was liquid butter fat or sunflower oil, and the continuous phase contained milk proteins. The low shear forces applied in membrane emulsification had a beneficial effect on the physicochemical and molecular properties of the proteins. [85] also prepared a low fat spread with a fat content of 25% (v/v). Membrane emulsification was proved to be suitable for preparation of water in oil food emulsions at large scale thanks to the scale-up ability of membranes and their high production rates.

In the last decade, several studies proposed the preparation of food grade emulsions or double emulsions using membrane emulsification. Rather than being industrial applications, these studies proposed formulations including components that can be used in the food industry. For example, [55] prepared food-grade double emulsions by premix membrane emulsification. The formulation consisted of beetroot juice as inner water phase, sunflower oil as oil phase and whey protein isolate solution as outer water phase. The

aim was to protect the water soluble pigments contained in the beetroot juice from degradation thanks to its encapsulation in the inner water phase. [111] proposed a membrane emulsification technique for the encapsulation of biophenols recovered from olive mill wastewaters. Catechol was chosen as a biophenol model and encapsulated in water-in-oil emulsions. The valorization of biophenols recovered from olive mill waste waters is particularly interesting. [79] prepared food grade $water_1$/oil/$water_2$ emulsions by membrane emulsification using microsieve membranes for the encapsulation of garlic extracts in the water phase. Garlic is known for its positive effects on human health with antimicrobial, antifungal, antiparasitic and antiviral activities. Its disadvantages, like instability, volatility and unpleasant taste and odor can be reduced by its encapsulation.

Other food industries could benefit of the advantages of the membrane emulsification techniques. For example, beverages such as flavored tea, flavored water, juice drinks, and dairy-based juice drinks [128] are based primarily on the essential oils from the peel of the fruits (e.g., orange oils, lemon oils). They are obtained by adding flavors from essential oils at a concentration lower than the soluble concentration or by preparing emulsions of these flavors (beverage emulsions) [113]. Membrane emulsification may be a possible process to produce beverage emulsions.

Conclusion

Membrane processes are intensively used in the food industry. The most common processes are filtrations (RO, NF, UF, and MF) for concentration or clarification of food suspensions like milk, fruit juices, wines, and beers. Several improvements have been proposed for the control of flow in membrane modules, reduction of fouling by new techniques, and optimization of chemical or physical cleaning. The advancements and understanding of UF and MF have also made possible fractionation of molecules of different sizes for example in the dairy industry. Other membrane processes include ED for the stabilization of wines and deacidification of fruit juices.

Several trends have emerged these last years in the use of membrane processes for food industries. Complete membrane plants are now available for different applications and commercialized by several companies. For example, food companies can invest in complete plants for milk sterilization, wine clarification, or beer dealcoholization. This also true for less traditional techniques, like membrane emulsification, for which complete plants are commercialized by several European and Asian companies. This confirms that a large number of membrane processes have reached a high degree of maturity.

Membrane processes are still under investigation to better understand and to reduce fouling, which is a main limitation in food applications where concentrated feeds contain a large range of compounds with different sizes, charges, hydrophobicities/hydrophilicities, etc. The emerging trends concern the in-line characterization of membrane fouling, advanced characterization techniques like HPLC coupled mass spectrometry, and advanced simulations methodology like molecular simulation [35, 116].

An increasing concern is also related to the environmental consequences of all processes used, with energy and water consumption, and waste water release. In that sense, all major processes and especially membrane processes are concerned. For example, the volume of waste water in the dairy industry is minimized and this effluent is treated to recover valuable compounds. In the beer industry, environmental issues are also of major concern and several membrane processes are proposed to treat effluents. Eco-conception of new membrane processes has also emerged as an important field. Cheese making by membrane techniques can be realized directly in the farms to optimize milk collection and limit transport [125]. The collection of fruit juices can be made in farms of tropical countries and their concentration realized in solar dryers with membrane pouches [110]. These trends should increase in the next few years in all major food industries.

New food products are also emerging as consumers are increasingly looking for healthy and natural products. Antioxidant, antiaging, etc., activities are requested. In this regard, membrane processes can help to develop new food formulations thanks to their ability to purify, concentrate, and also elaborate new products.

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Compliance with Ethical Standards

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