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Review Paper

A Comprehensive Review of Membrane Distillation and Osmotic Distillation in Agro-Food Applications

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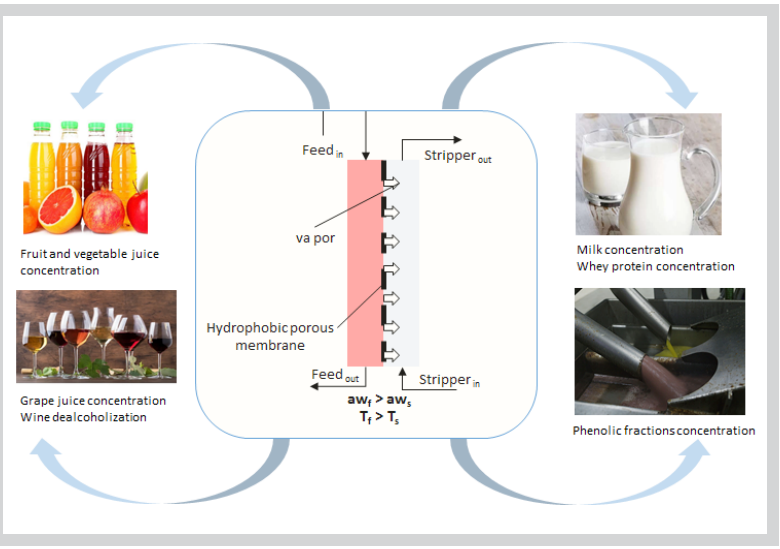
Highlights

- An overview of MD and OD in agro-food processing is presented
- Fruit and vegetable juice concentration
- Grape juice concentration and wine dealcoholization
- Concentration of milk and whey proteins
- Concentration of phenolic fractions from agro-food wastewaters

Graphical abstract



Abstract

Membrane distillation (MD) and osmotic distillation (OD) are emerging athermal processing techniques of great interest in agro-food production where the most part of products is sensitive to thermal treatments. With respect to conventional methods, MD and OD are competitive alternatives, able to work in an environmental-friendly and cost-efficient way, for preserving the nutritional and sensorial attributes of processed foods, in agreement with the increased expectations of consumers and producers. This review will provide an overview of the current status and recent developments in the use of MD and OD in agro-food applications. Theoretical aspects and specific applications in the field of fruit juice concentration, milk and dairy industry, wine dealcoholization and agro-food waste processing, are presented and discussed. The integration of these processes with other membrane operations within the logic of the process intensification strategy is also evaluated in order to overcome specific challenges for a sustainable industrial development.

Contents

1. Introduction

Membrane separation technologies have attracted much attention in the food processing industries over the last years. After water treatment, the food industry represents a significant part of the turnover of the membrane manufacturing industry worldwide with well-established applications in dairy

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(milk and whey), fruit juice (apple, pear, grape, citrus), fermented products (wine, beer, vinegar), animal products (gelatin, eggs), plant proteins (soy) and sugars (dextrose, sucrose, dextrins, high-fructose syrup) processing.

Pressure-driven membrane operations, such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) are consolidated systems for clarification, concentration, fractionation, desalting, recovery and purification of target compounds in different areas of the food production, with several advantages over conventional methodologies including high selectivity, no thermal stress of processed fluids due to moderate operating temperatures, no use of chemical additives, easy scale-up, compact and modular design, low energy consumption [1]. These processes are less energy intensive when compared with thermal evaporation (14.36 kJ per kg water removed against 300 kJ of thermal processes) and freeze concentration. However, concentration polarization, membrane fouling, shear damage and constraints on the maximum achievable concentration are typical drawbacks limiting their application in liquid foods concentration. Membrane distillation (MD) and osmotic distillation (OD) are emerging technologies which have attracted a growing interest in recent years for the concentration of food products, including dairy products and fruit juices [2,3]. Both processes are driven by a vapor pressure difference between porous hydrophobic membrane surfaces, through which only water vapor molecules can pass. Therefore, concentration polarization is not a limiting factor and high solid contents can be achieved.

The full commercial application of these processes is still limited by low fluxes and higher production costs than thermal evaporation. However, their combination with conventional membrane operations can provide a very attractive approach to improve the product quality and process economics.

Based on current literature reports, the present paper provides the most recent development studies on the use of MD and OD in agro-food applications, including the concentration of fruit and vegetable juices, dehydration of milk and whey, concentration of grape must and wine dealcoholization, concentration of valuable compounds from olive mill wastewaters. Technological inputs arising from the combination of MD and OD with pressure-driven membrane operations are also analysed and discussed in order to overcome typical drawbacks and to improve the performance of these processes.

2. Fundamentals

2.1. Osmotic distillation

Osmotic distillation (OD) is a non-pressure driven membrane processes, also known as isothermal membrane distillation, membrane evaporation and osmotic evaporation, which can be used to extract water from aqueous solutions under atmospheric pressure and room temperature, thus avoiding thermal degradation of the solutions [2]. In this process a hypertonic salt solution (stripping solution) is used to promote a vapor transfer across the pores of a microporous hydrophobic membrane from the high-vapor pressure phase to the low one: water evaporates at the dilute vapour-liquid interface, diffuses through the membrane pores and condenses at the membrane/brine interface on the surface of the solution with lower vapor pressure (Figure 1) [4,5].

The water vapor pressures at the pore mouths are related to the temperature and activities prevailing in the liquids facing the membrane by:

$$P_{w1} = P^*_{w1} \cdot a_{w1} \quad (1)$$

$$P_{w2} = P^*_{w2} \cdot a_{w2} \quad (2)$$

in which P^*_w represents the vapor pressure of pure water and a_w the water activity in the solutions. The driving force ($\Delta P_w = P_{w1} - P_{w2}$) for water transport is sustained by the activity difference $\Delta_{aw} = a_{w1} - a_{w2}$.

The hydrophobic nature of the membrane prevents penetration of the pores by aqueous solutions, creating air gaps within the membrane. A maximum critical pore size exists at which the liquid penetrates the microporous hydrophobic phase, and for a given pore size r_p, a critical penetration pressure P_c can be defined by the Laplace equation:

$$P_c = \frac{2\gamma \cdot cos\theta}{r_p} \quad (3)$$

The water transport that relates the mass flux (J) to the driving force is given by:

$$J = K \cdot \frac{\Delta P_w}{p_{air}} \quad (4)$$

where p_{air} is the mean pressure of air entrapped into pores and K the overall mass transfer coefficient which accounts for all three resistances for water transport (feed, membrane and brine side).

Since water transport involves condensation and evaporation phenomena, a temperature gradient through the membrane is generated, even if bulk temperatures of solutions separated by the membrane are equal. Consequently, a heat transfer tending to reduce the driving force for the water transport should be considered in addition to a mass transfer [6].

The total heat transferred across the membrane (Q) is given by:

$$Q = H \cdot \Delta T \quad (5)$$

where H is the overall heat transfer coefficient which accounts for all the resistances (feed, membrane and stripping solution) [7].

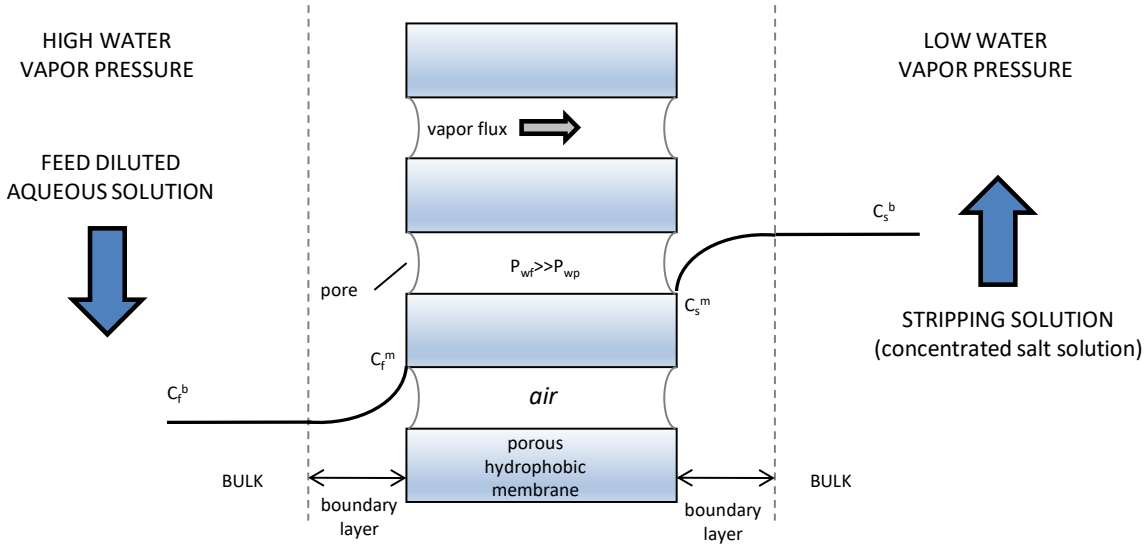

Fig. 1. Schematic representation of osmotic distillation (C_f^b, solute concentration in the bulk of the feed side; C_f^m, solute concentration at the membrane interface of the feed side; C_s^b: concentration of the stripping component in the bulk; C_s^m, concentration of the stripping at the membrane interface).

Osmotic agents used in OD should be thermally stable and also preferably no-toxic, no-corrosive and low cost. They should also have high osmotic activity in order to maintain a lower vapor pressure and to maximize the driving force. NaCl and $CaCl_2$ are commonly used at this purpose, although they are substantially more corrosive toward stainless steel than other osmotic agents. Potassium salts of ortho- and pyro-phosphoric acid offer several advantages, including low equivalent weight, high water solubility, steep positive temperature coefficients of solubility and safe use in foods and pharmaceuticals [8].

Microporous membranes used in OD are typically hydrophobic in nature; polymers with low surface free energy such as polyethylene (PE), polytetrafluoroethylene (PTFE), polypropylene (PP), and polyvinylidene fluoride (PVDF) are the most commonly preferred polymeric membranes due to their low surface tension values. Membranes should have a low resistance to mass transfer, low thermal conductivity to prevent heat loss across the membrane, high liquid entry pressure of water to maintain dry membrane pores and good chemical resistance.

The pore sizes are usually between 0.2 and 1.0 μm. Membrane porosity ranges from 60% to 80% and the overall thickness is of about 80-250 μm, depending on the absence or presence of support. By referring to the membrane configuration, hollow fiber membranes are generally preferred over flat-sheet and spiral-wound membranes.

2.2. Membrane distillation

In MD the water vapour transfer is promoted by a vapour pressure difference between two sides of a microporous hydrophobic membrane which prevents penetration of the pores by aqueous solutions due to surface tensions, unless a transmembrane pressure higher than the membrane liquid entry pressure is applied. However, in MD the physical origin of the vapour pressure difference is a temperature gradient rather than a concentration gradient: the feed is maintained at high temperature while cold water is used as a stripping permeate. Therefore, MD is a thermal-driven process (Figure 2).

Feed temperatures in MD are typically in the range 60-90 °C, although temperatures as low as 30°C have been used. Operating pressures are generally of about 0-1 bar, hence much lower than conventional pressure-driven membrane processes such as RO. Consequently, lower equipment costs and increased process safety can be achieved. Furthermore, the mechanical resistance of the membrane is greatly reduced.

Since MD operates on the principles of vapour-liquid equilibrium, another advantage, in comparison with traditional pressure-driven membrane processes, is represented by its high rejection (theoretically 100%) towards ions, macromolecules, colloids, cells and other non-volatile compounds [9].

As in the OD process the main drawback of the MD process is the risk of wetting of the hydrophobic membrane with a consequent reduction of permeate flux, in case of partial pore wetting, or permeate quality deterioration as the consequence of full wetting [10].

Therefore, the process solutions must be aqueous and sufficiently dilute. This limits MD to applications such as desalination, removal of trace volatile compounds from wastewater and concentration of non-volatile aqueous solutions.

The most common configuration of MD used for the concentration of liquid foods is the direct contact membrane distillation (DCMD) in which both feed and the permeate liquid are in direct contact with the membrane in their respective compartments. Volatile molecules evaporate at the hot liquid/vapour interface, cross the membrane pores in the vapour phase and condense on the cold liquid/vapour interface inside the membrane module [11]. In the vacuum membrane distillation (VMD) vacuum is applied on the permeate side of the membrane by means of a vacuum pump and condensation takes place outside the membrane module [12].

In the air gap membrane distillation (AGMD) a condensing surface is separated from the membrane by an air gap. Volatile molecules cross both the membrane pores and the air gap and finally condense over a cold surface inside the membrane module. In the sweeping gas membrane distillation (SGMD) a cold inert gas sweeps the permeate side of the membrane carrying the volatile molecules. In this case, condensation occurs outside the membrane module [13].

Schematics of MD configurations are illustrated in Figure 3.

As for OD, polymeric membranes used in MD are typically hydrophobic in nature. They are realized in flat-sheet or capillary configurations by phase-inversion, stretching of dense films and thermally induced phase separation. Typical commercial polymeric membranes used in MD are summarized in Table 1.

Several authors have investigated the improvement of hydrophobic properties of MD membranes employing novel materials, or applying surface modification through manipulating surface chemistry and surface geometry by nanoparticle coating and surface fluorination [14-16].

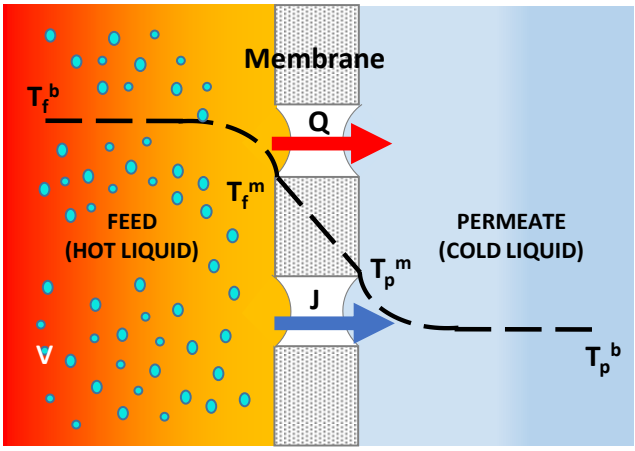

Fig. 2. Schematic representation of membrane distillation (T_f^b, feed temperature in the bulk; T_f^m, feed temperature at the membrane interface; T_p^b, permeate temperature in the bulk; T_p^m, permeate temperature at the membrane interface).

Efforts have been made also in order to avoid wetting and reduce temperature polarization phenomena. For example, the use of net-like turbulence promoters (spacers) has been proposed in order to change the flows dynamic of the recirculating streams and to promote regions of turbulence which enhance process flux [17]. Promising results for wetting inhibition have been achieved through the use of PTFE superhydrophobic membrane in combination with air flow and a mesh spacer on the feed side. The presence of air bubbles inhibited the occurrence of wetting even for high concentrations of the surface-active species (up to 0.8 mM SDS) in the feed solution [18].

Polymeric nanofibers prepared by the electrospinning technique are innovative candidates for the MD process [19]. In particular, PVDF nanofiber membranes prepared and used for DCMD have shown high performance in terms of water flux (25-31 kg/m^{2}h) and salt rejection (greater than 99.9%) when employed in water desalination [20,21].

Reduced graphene oxide/poly(vinylidene fluoride-co-hexafluoropropylene) (rGO/PVDF-HFP) flatsheet membranes for use in DCMD have been recently prepared by Chen et al. [22] through the electrospinning technique. The prepared membranes exhibited excellent stability and durability with a salt rejection of over 99.97% and an average flux of 20.37 kg/m^{2}h, a maximum water contact angle of 139° and an increased liquid entry pressure (LEP) of up to 103.42 kPa. The pore size distribution was in the range of 0.20-0.92 μm with a desired mean pore size compared with the pristine PVDF-HFP membranes. Graphene consists of a 2D sheet of sp2-bonded carbon atoms in hexagonal honeycomb lattices acting as effective sorption sites for water vapor, while repelling water molecules and salt ions (Figure 4).

Ceramic membranes have attracted a great interest in the last years for a wide array of membrane-based separation applications including MD [23]. These membranes are generally hydrophilic in nature; therefore, their surface has to be modified to obtain hydrophobic properties. The introduction of hydrophobic chemical groups of silane agents (i.e. fluoroalkylsilane) into the membrane surface through the formation of hydrogen, ionic, van der Waals or covalent bondings is the most common method used for ceramic membrane hydrophobization. Silane agents grafting approaches include immersion [24], chemical vapor deposition [25] and the mixing of silane agent in sol-gel [26]. Among them the grafting via immersion offers greater advantages in terms of process duration, energy consumption, production cost, membrane structure and stability of the hydrophobic layer.

Stainless steel membranes, typically used in microfiltration, modified by depositing on their surface a very thin film of silicone compounds, have been also tested for MD operations [14].

Table 1
Typical commercial membranes used in MD applications.

Membrane Type	Manufacturer	Configuration	Material	Thickness (μm)	Porosity (%)	Average pore size (μm)
TF200	Gelman	Flat sheet	PTFE/PP	178	80	0.2
TF450			PTFE/PP	178	80	0.45
TF1000			PTFE/PP	178	80	1.0
Taflen			PTFE	60	50	0.8
Metricel			PP	90	55	0.1
FGLP	Millipore	Flat sheet	PTFE/PE	130	70	0.2
FHLP			PTFE/PE	175	85	0.5
FALP			PTFE/PE	150	85	1.0
Durapore GVHP			PVDF	110	75	0.22
Durapore HVHP			PVDF	140	75	0.45
Celgard 2500	Hoechst Celanese Co.	Flat sheet	PP	28	45	0.07
Celgard 2400		Flat sheet	PP	25	38	0.05
Celgard X-20		Capillary	PP	25	35	0.03
PTS 20	Gore	Flat sheet	PTFE/PP	184	44	0.2
PT 20			PTFE	64	90	0.2
PT 45			PTFE	77	89	0.45
G-4.0-6-7	GoreTex Sep.	Flat sheet	PTFE	100	80	0.20
3MA	3M Corporation	Flat sheet	PP	91	66	0.29
3MB			PP	81	76	0.40
3MC			PP	76	79	0.51
3MD			PP	86	80	0.58
3ME			PP	79	85	0.73
	Teknorama	Flat sheet	PTFE	-	80	0.2
			PTFE	-	80	0.5
			PTFE	-	80	1.0
Accurel S6/2 MD020TP2N	Akzo Nobel Microdyn	Capillary	PP	450	70	0.2
MD020TP2N	Enka Microdyn	Capillary	PP	1550	75	0.2
Accurel BFMF 06-30-33	Enka A.G. Euro-Sep	Capillary	PP	200	70	0.2
Sartocon-Mini SM	Sartorius	Capillary	Polyolefine	-	-	0.22

Legend: PP, polypropylene; PTFE, polytetrafluoroethylene; PVDF, polyvinylidene fluoride; PE, polyethylene.

3. Applications

3.1. Concentration of fruit and vegetable juices

Fruit juices, owing to their valuable nutritional profile, are one of the most widely traded food products in the world. Juice concentration is one of the basic unit operations of fruit juice production in which the solids content of the juice is increased from 10% to 12% up to 65-75% by weight [27]. This step is mainly accomplished in order to i) reduce water activity of the juice product which lengthens its shelf life; ii) minimize packaging, storage, and transport costs, and (iii) stabilize or simplify the handling of the final juice product [28].

Commercial concentration processes usually involve the use of multi-stage vacuum evaporation in which the juice is boiled in a sequence of stages, each held at a lower pressure than the last or occupying a larger surface area. In most cases the volatile components are recovered and added back to the concentrated product. Despite its economic feasibility, thermal evaporation exhibits some disadvantages when applied to fruit juices such as heat degradation of sensorial and nutritional characteristics with partial loss of aroma and nutrients, induction of cooked taste due to furfural formation and browning due to Maillard-reactions [29]. Unlike heat evaporation, cryoconcentration has no or little effect on taste, aroma, color, or nutrients of juice products. However, the degree of achievable concentration is lower than thermal evaporation. High vapital costs, large energy consumption, difficult control of ice crystal growth for a longer time and solids loss due to juice entrapped in ice crystals are additional drawbacks [30].

Product quality improvement and energy savings have guided the development of *minimal* processing techniques. Membrane concentration processes, such as MD and OD present some attractive potentials to overcome limitations associated with conventional technologies [31,32]. These processes can be used to selectively extract water from aqueous solutions under atmospheric pressure and at room temperature, thus avoiding thermal and mechanical damage of the solute [4,33]. Therefore, fixed costs and mechanical requirements on the membrane are greatly reduced due to lower pressure requirements.

Evaporation fluxes in MD are affected by operating parameters (feed flow rate, feed temperature, feed concentration, permeate flowrate and permeate temperature) as well as by membrane properties [3]. PVDF membranes with a nominal pore size of 0.1 □m showed a total retention of orange juice compounds such as sugars and organic acids [34]. These

membranes showed a very good retention of orange juice compounds such as soluble solids, sugars and organic acids, with a 100% rejection of sugars and organic acids. Evaporation fluxes increased by increasing the feed juice temperature for each flowrate at a constant inlet temperature of the cooling water. Similarly, permeate flux increased by increasing the feed flowrate through the reduction of fouling phenomena due to the deposition of pectin and cellulose on the membrane surface.

Halar fibers (copolymer of ethylene and chlorotrifluoroethylene) exhibited a better performance in terms of water removal and energy saving when compared with PVDF fibers in the removal of water from glucose solutions [35]. Evaporation fluxes were of about 5.8 kg/m^{2}h when concentrating 30% glucose solution at 40 °C.

Recently hydrophobic alumina hollow fiber membranes were prepared by using a combined phase inversion and sintering method and then used in a VMD system for sucrose concentration [36]. Sucrose solutions were concentrated from 10 °Brix up to 50 °Brix at a feed flowrate of 5 L/min and temperature of 70 °C with average permeate fluxes of about 27 kg/m^{2}h.

Steady-state evaparation fluxes of about 10 kg/m^{2}h were obtained in the concentration of clarified sugarcane juice at 20 °Brix by using a PP flat-sheet membrane at constant ΔT (50 °C) and feed flowrate (1000 mL/min) [37].

Highly concentrated apple juice (64 °Brix) was produced by DCMD by using a PP hollow fiber membrane module (ENKA MD-020-2N-CP, Microdyn) with a tube and shell configuration. Evaporation fluxes, in the range of 1-2 kg/m^{2}h, increased by increasing the temperature gradient through the membrane (mantaining the feed temperature at 32 °C) as well as by increasing both feed and distillate flowrates in the intra- and extra-fiber volumes, respectively [38]. The juice viscosity at high concentration induced severe polarization phenomena. However, temperature polarization resulted higher than concentration polarization.

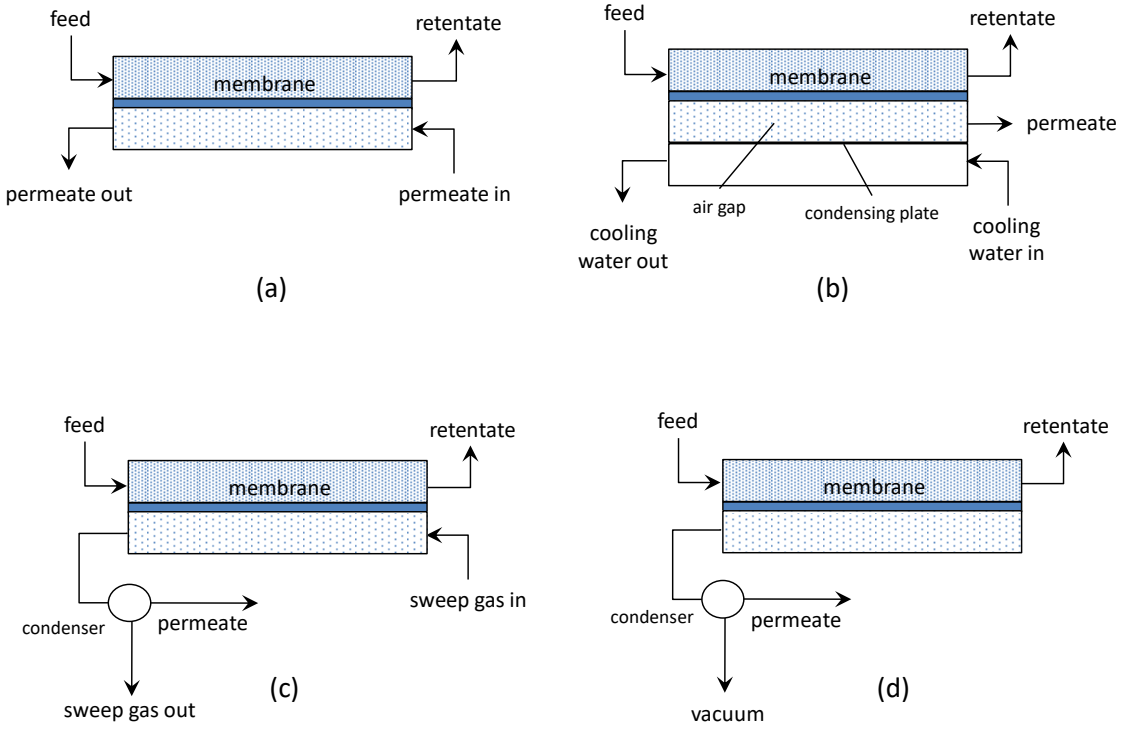

Fig. 3. Membrane distillation configurations: a) Direct contact membrane distillation (DCMD); b) air gap membrane distillation (AGMD); c) sweep gas membrane distillation (SGMD); d) vacuum membrane distillation (VMD).

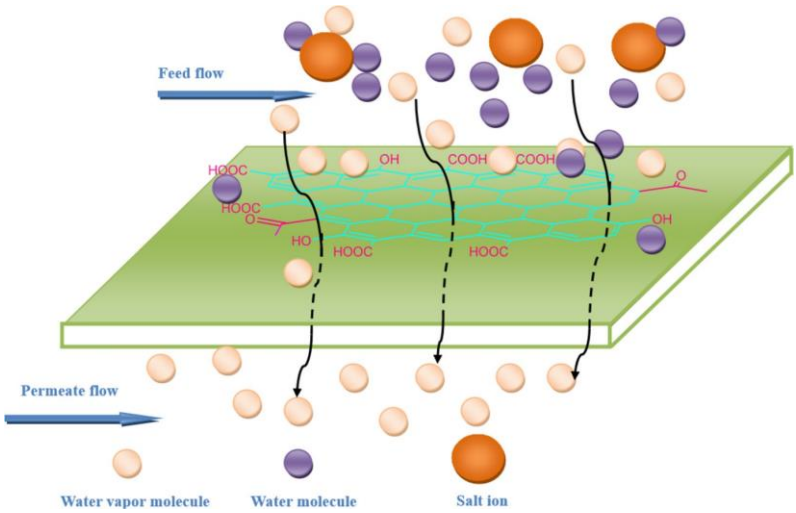

Fig. 4. Schematic of proposed mechanism for PVDF-HFP-rGO membrane [22].

Like MD, OD has a great potential for concentrating fruit juices at high total soluble solids value under mild operating condition and without product damage. The low operating pressure results in lower energy consumption and capital investment reduced fouling phenomena and possibility to use membranes characterized by lower mechanical resistance in comparison with pressure-driven membrane processes. Evaporation fluxes are mainly affected by feed and osmotic agent flow rate as well as by the osmotic agent concentration. Flowrates directly affect the thickness of the boundary layer at the membrane surface that presents a resistance to the mass transfer; the brine concentration affects the vapor pressure gradient through the membrane, which is directly related with magnitude of the driving force [4,39]. The contribution of concentration polarization on transmembrane flux is more prominent when compared to that of temperature polarization [5].

Typical applications of OD in the concentration of fruit and vegetable juices are listed in Table 2.

The most well-known module designed for concentration-driven mass transfer in OD is the Liqui-Cel™ Extra-Flow membrane contactor manufactured by 3M (Charlotte, NC; formerly Hoechst Celanese) [69] depicted in Figure 5. This module uses Celgard® microporous polypropylene hollow fibers that are woven into a fabric and wrapped around a central tube feeder that supplies the shell side fluid. These fibres are approximately 0.3 mm in external diameter with a wall thickness of about 0.03 mm, a mean pore diameter of about 30 nm and a porosity of about 40%. In most applications for fruit juice concentration the juice is recirculated in the shell side of the module, while the stripping solution is recirculated in a counter current mode through the lumen side.

Hollow fiber membrane contactors are preferred over flat sheet membranes due to their high specific area per unit volume, easy scale-up and low manufacturing cost [63].

Recently, Rehman et al. [64] evaluated the effect of fouling on the performance of PP hollow fiber membranes in the OD concentration of pomegranate juice by using a Liqui Cel® 1.7 × 5.5 in. mini module. Scanning Electron Microscopy (SEM) analysis revealed a thick and continuous fouling layer on the membrane surface that can be linked to a combination of the leftover suspended particles and the organic layer including sugars such as glucose, sucrose and fructose, anthocyanins, polyphenols and organic acids such as gallic, ellagic, chlorogenic, caffeic, citric and oxalic acids.

Almost continuous fouling layers were also observed on the surfaces of PP and PVDF membranes in the OD of apple and beet juices [70]; for PTFE membranes the surface showed randomly distributed clean patches that remained uncovered by the fouling layer. The porosity of all tested membranes was reduced after the OD process from 7% (for PVDF membranes) to 37% (for PP membranes). Previously, Durham and Nguyen [71] found that the the surface tension of PTFE membranes used in the OD concentration of tomato puree was reduced due to the adherence of red and yellow tomato pigments which increased the fouling propensity and decreased evaporation fluxes.

Recently, nanofibrous polyether-block-amid (PEBA) membranes prepared by the electrospinning technique have been tested for the concentration of pomegranate juice by OD using calcium chloride dehydrate as stripping solution [72]. Aroma and phenolic compounds were better preserved in comparison to thermal evaporation producing a high quality concentrated juice. According to the economic analyses, the profit of the OD process resulted lower than that of thermal evaporation due to the higher equipment costs of the OD process (a total membrane surface area of 328 m^2 was estimated). Moreover, the break-even point of the OD process was higher than that of the evaporation process.

Physico-chemical, biochemical and aromatic qualities of several raw juices and vegetable extracts concentrated by OD, including black mulberry juice [73], cranberry juice [74], roselle extract [55], noni juice [53] and pineapple juice [40] resulted well preserved in comparison to thermally evaporated samples.

The performance of both MD and OD processes in the concentration of fruit juices can be improved significantly through a preliminary clarification step aimed at removing suspended solids and pectins from the raw juice and decreasing the juice viscosity. Microfiltration (MF) and ultrafiltration (UF) membranes are useful approaches for this purpose as an alternative to conventional clarification steps based on the use of fining agents.

UF membranes with nominal pore size of 0.1 μm or less produced an appreciable enhancement of the evaporation flux in the concentration of grape juice by OD in comparison with the untreated juice [75]. The UF process resulted also in an increase in the juice surface tension with a consequent reduction in membrane wetting. Similarly, the use of 30 kDa PVDF membranes removed macromolecular particles from pomegranate juice preventing membrane wetting during the subsequent OD concentration process [61]. Significant improvements of evaporation fluxes were also observed in the concentration of pineapple juice by OD and single-strength orange juice by MD after a UF pre-treatment [34,52]. The influence of the apple juice pre-treatment before concentration by MD was studied by Lukanin et al. [76]. After a fermentation process, the apple juice was submitted to an enzymatic treatment with protease, followed by a clarification step by UF. This process allowed to remove biopolymers and proteins resulting in increased transmembrane flux during the MD process.

Integrated membrane processes based on the use of MF or UF for juice clarification followed by MD or OD for juice concentration have been investigated on both laboratory and pilot scale for several fruit and vegetable juices including apple [54,77], orange [45,78,79], kiwifruit [42,48], cactus pear [49], camu-camu [43], bergamot [58], pomegranate [56,64], passion fruit [59], cholupa [60], pineapple [40], black currant [80], melon [44], broccoli [68], chokeberry, redcurrant and cherry [51] juices. The investigated processes allow to obtain high levels of soluble solids (up to 65-68 °Brix) with evaporation fluxes in the range of 1-10 kg/m^2h and without modifying the main physico-chemical parameters of the clarified juice.

In Table 3 the physico-chemical parameters of pomegranate juice clarified and concentrated by integrated UF/OD process are reported. The clarified juice was concentrated from 16.2 to 52 °Brix by using a Liqui Cel® Extra-Flow 2.5 x 8-in. membrane contactor and 10.2 mol/L calcium chloride dehydrate solution as osmotic agent [56]. A 3.2-fold concentration of total soluble solids was achieved by using OD without any back diffusion of solute and burned off flavor due to the hydrophobic nature of the membrane. Organic acids were well preserved, while anthocyanins and total antioxidant activity were reduced of 23% and 4% in comparison to the clarified juice. On the other hand, a strong degradation of anthocyanin pigments was observed in pomegranate juice samples concentrated by thermal evaporation accomplished by the presence of significant levels of 5-hydroxymethyl furfural (an indicator of potential browning of the juice) and by the reduction of minerals, such as sodium, iron, lead, copper and zinc between 44% and 69% [81]. Similarly, the orange juice concentrated by a two-step DCMD process up to 65 °Brix, after a preliminary UF step, still showed a high TAA value (about 6.6 mM Trolox) when compared with the raw juice and the clarified juice (6.52 and 6.40 mM Trolox, respectively), confirming the validity of the process in preserving the original quality of the fresh juice [79].

Figure 6 shows the performance of an OD Liqui Cel® Extra-Flow 2.5 x 8-in. membrane contactor in the concentration of clarified cactus pear juice (recirculated in the shell side of the module) up to 61 °Brix by using calcium chloride dehydrate as stripping solution (recirculated within the lumen side). In the first part of the process (0-175 min) the evaporation flux decay can be attributed mainly to the dilution of the brine solution. A more rapid decline of the evaporation flux, due to the exponential increasing of the juice viscosity, can be observed starting from a total soluble solids (TSS) content of 33 °Brix. The break-even point of the viscosity curve with respect to concentration corresponds to this TSS value [49]. As reported also by other Authors, at higher TSS values the OD flux depends mainly on juice viscosity and, consequently, on juice concentration and temperature [7,82].

The large amount of water present in the initial juice promotes a fast brine dilution, which negatively affects the productivity of the OD process. For this purpose, several Authors investigated the use of reverse osmosis (RO) as a pre-concentration step before a final concentration of the juice by OD. In particular, the use of UF or MF, RO and OD in a sequential design has been proposed for several fruit and vegetable juices including carrot [41], orange [83], mandarin [67], acerola [84], blackcurrant [50], apple [57] and pomegranate [66] juices.

The concentrated orange juice produced through a combination of UF, RO and OD processes was characterized by a slight decrease of the total antioxidant activity (TAA) (~15%) in comparison to the fresh juice which was attributed to a partial degradation of ascorbic acid (~15%) and anthocyanins (~20%) probably on account of the high pressure (50 bar) experienced by the juice during the RO treatment. No significant variations were observed for hydroxycinnamic acids and for flavanones, which appeared to be very stable under the selected operating conditions of the RO process. On the other hand, a higher degradation of bioactive compounds and TAA was observed for thermally concentrated juice (TAA, ~26%; ascorbic acid, ~30%, anthocyanins, ~36%) [83].

The combination of NF and RO operations was proposed by Sotoft et al. [85] as preconcentration step of blackcurrant juice (up to 45 °Brix) before a final concentration by DCMD. This approach allows to exploit the high rejection of RO membranes and the high concentration factor of NF membranes in order to overcome the high osmotic pressure limitations typically encountered in RO. The NF permeate is recirculated back to the RO unit, while the retentate stream is submitted to the DCMD unit where the juice is concentrated juice up to 65-70° Brix (Figure 7). The production of the proposed system was fixed at 17,283 ton of 66 °Brix concentrated juice/year with a production price of 0.40 €/kg (assuming a membrane lifetime of 1

year). The estimated operation cost resulted 43% lower than that of conventional evaporators.

Table 2
Concentration of fruit and vegetable juices by osmotic distillation (OD).

Juice	Osmotic agent	Membrane type	Average flux (kg/m^2h)	Reference
Pineapple (clarified by MF)	$CaCl_2$ 4.6 m	hollow fiber, PP	n.r.	[40]
Citrus and carrot (clarified by UF and preconcentrated by RO)	$CaCl_2x2H_2O$ 60-66% w/w	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.8 (carrot juice); 0.8 (blood orange juice)	[41]
Kiwifruit (clarified by UF)	$CaCl_2x2H_2O$ 60% w/w	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.5	[42]
Camu-camu (clarified by MF)	$CaCl_2$ 4.0-5.2 M	plate and frame, thin PTFE layer sealed on a PP supporting net (TF200 Pall-Gelman)	10	[43]
Melon (clarified by MF)	$CaCl_2$ 5.3-5.6 M	hollow fiber, PP	0.6	[44]
Orange (clarified by MF)	$CaCl_2$ 5.5 M	hollow fiber, PP	0.65	[45]
Sucrose, apple	$CaCl_2$ 3.5-6 M	tubular, PP (Microdyn)	-	[46]
Orange, sucrose	$CaCl_2x2H_2O$ 4.9 M	Hollow fiber, PP (Accurel® PP Q/32, Membrana GmbH)	0.9 (orange juice); 1.1 (sucrose)	[47]
Kiwifruit (clarified by UF)	$CaCl_2x2H_2O$ 60% w/w	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.8	[48]
Cactus pear (clarified by UF preconcentrated by RO)	$CaCl_2x2H_2O$ 60% w/w	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.6	[49]
Blackcurrant (clarified by MF and preconcentrated by RO)	$CaCl_2x2H_2O$, 65°Brix (laboratory scale); $CaCl_2$ 60.7°Brix (large scale)	capillary, PP (MD 020 CP 2N, Microdyn) (laboratory scale); capillary, polypropylene (MD 150 CS 2N, Microdyn) (large scale)	0.7 (laboratory scale); 0.6 (large scale)	[50]
Chokeberry, redcurrant and cherry (clarified by UF)	$CaCl_2$ 6 M	tubular, PP (Microdyn)	4.0	[51]
Pineapple (single strength and clarified by MF)	$CaCl_2$ 5.5-6 M	flat-sheet, thin PTFE layer sealed on a PP supporting net (TF200 Pall-Gelman)	10.5 (single strength juice); 11 (microfiltered juice)	[52]
Noni (*Morinda citrifolia*)	$CaCl_2$ 6 M	hollow fiber, PP (Liqui Cel® minimodule 1.7 × 5.5 in., Membrana);	0.09	[53]
Apple (clarified by UF)	$CaCl_2x2H_2O$ 65% w/w	capillary, PP (MD 020 CP 2N, Microdyn)	n.r.	[54]
Roselle extract, apple and grape	$CaCl_2$ 5.5-6 M	hollow fiber, PP	1.05 (roselle extract); 1.05 (grape juice); 1.2 (apple juice)	[55]
Pomegranate (clarified by UF)	$CaCl_2x2H_2O$ 10.2 M	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.5	[56]
Apple (clarified by UF and preconcentrated by RO)	$CaCl_2$ 5.5 M	flat-sheet, thin PTFE layer sealed on a PP supporting net (TF200 Pall-Gelman)	0.75	[57]
Bergamot (clarified by UF)	$CaCl_2x2H_2O$ 10.2 M	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.9	[58]
Passion fruit (clarified by UF)	$CaCl_2$ 45%w/v	capillary, PP (MD 020 CP 2N, Microdyn)	0.52	[59]
Cholupa (*Passiflora maliformis*) (clarified by UF)	$CaCl_2$ 45%w/v	capillary, PP (MD 020 CP 2N, Microdyn)	0.65	[60]
Pomegranate (clarified by UF)	$CaCl_2x2H_2O$ 65% w/w	hollow fiber, PP (MD 020 CP 2N, Microdyn)	1.1	[61]
Orange press liquor (clarified by UF, preconcentrated by NF)	$CaCl_2x2H_2O$ 10.2 M	hollow fiber, PP (Liqui-Cel® Extra-Flow 2.5 × 8-in. membrane contactor, Hoechst-Celanese)	0.7	[62]
Sucrose, apple and orange	$CaCl_2$ 5 M	hollow fiber, PP (Liqui Cel® contactor module X-50, Membrana)	0.35 (sucrose, feed temperature 35°C); 0.18 (apple, feed temperature 30°C); 0.081 (orange, feed temperature 30°C)	[63]
Pomegranate (clarified by UF)	$CaCl_2$ 6 M	hollow fiber, PP (Liqui Cel® minimodule 1.7 × 5.5 in., Membrana)	0.62	[64]
Pomegranate (clarified by polypropylene spun filter)	$CaCl_2x2H_2O$ 6 M	flat-sheet, PTFE and PVDF (TS Filter Membranes)	0.7 (PVDF); 1.5 (PTFE)	[65]
Pomegranate (clarified by UF and preconcentrated by plasma modified RO membranes	$CaCl_2x2H_2O$ 65% w/w	capillary, PP (MD 020 CP 2N, Microdyn)	0.65	[66]
Nagpur mandarin (*Citrus reticulata* Blanco) (clarified by UF, preconcentrated by RO)	$CaCl_2x2H_2O$ 56.9% w/w	hollow fiber, PP (Liqui Cel® contactor module X-50, Membrana)	0.1	[67]
Broccoli (B. oleracea var. Italica) (centrifuged, clarified by UF)	$CaCl_2x2H_2O$ 65% w/w	capillary, PP (MD 020 CP 2N, Microdyn)	0.7	[68]

Legend: PP, polypropylene; PTFE, polytetrafluoroethylene; PVDF, polyvinylidene fluoride.

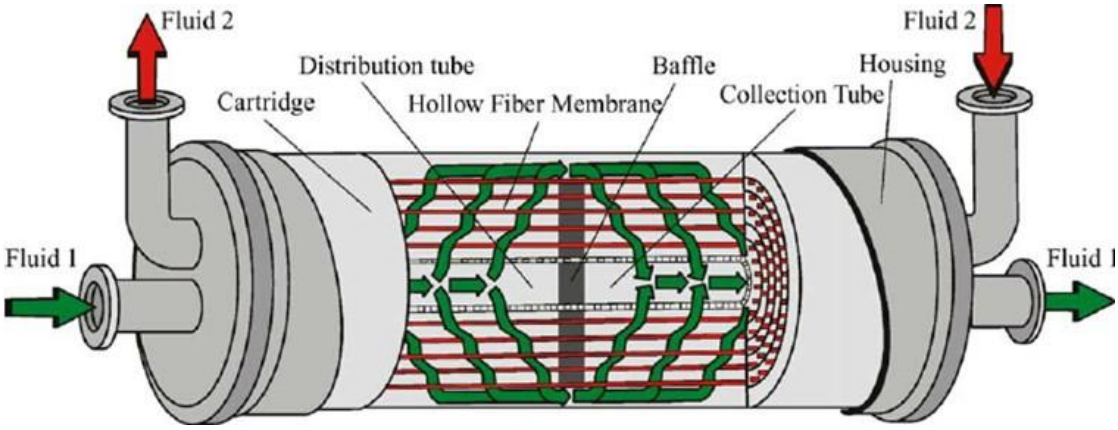

Fig. 5. The Liqui-Cel™ Extra-Flow membrane contactor [69].

Table 3
Physico-chemical properties of pomegranate juice clarified and concentrated by integrated UF/OD process (adapted from [56]).

	Fresh juice	Clarified juice	Concentrated juice
Total soluble solids (°Brix)	16.2	16.2	52.0
Suspended solids (%w/w)	4.8	n.d.	n.d.
Total acidity (g/L^{-1})	0.41	0.35	-
Ascorbic acid (mg/L)	68.0	47.0	44.0*
Malic Acid (mg/L)	1.9	1.82	1.80*
Citric acid (mg/L)	1.47	1.45	1.26*
Cyanidin 3,5-diglucoside (mg/L)	46.9	44.2	39.0*
Delphinidin 3-glucoside (mg/L)	20.6	17.7	11.5*
Cyanidin 3-glucoside (mg/L)	30.4	25.8	20.9*
Pelargolidin 3-glucoside (mg/L)	5.0	4.2	4.4*
Total antocyanins (mg/L)	102.8	90.7	75.85*
Total polyphenols (g catechin/L)	1.57	1.31	1.22*
Total antioxidant activity (mM trolox)	12.9	10.6	10.1*

*value referred to a TSS content of 16.2 °Brix

Cassano et al. [62] investigated the potential of an integrated membrane process for the recovery and concentration of flavonoids from orange press liquor, a citrus by-product enriched in bioactive compounds, such as flavonoids and phenolic acids, recognized for their beneficial implications in human health due to their antioxidant activity. The press liquor was previously clarified by UF and then preconcentrated by NF with a PES spiral-wound membrane (NF-PES 10, 2440C, from Microdyn-Nadir). Most of flavanones and anthocyanins were retained by the NF membrane (rejections of 97.4% and 98.9%, respectively) with a production of a permeate stream containing sugars and minerals. The final treatment of the NF retentate by OD produced a concentrated fraction of potential use in food and pharmaceutical industries.

Significant improvements of evaporation fluxes in OD can be achieved by combining the OD process with MD: the resulting process named as osmotic membrane distillation (OMD) results more effective than MD and OD alone.

Red fruits juices such as chokeberry, redcurrant and cherry were concentrated by OMD up to 62-65 °Brix after a preliminary clarification by UF [51]. The clarification step improved the efficiency of the OMD process, providing a less viscous feed stream with significantly lower fouling behavior during the concentration, at the same time excluding the possibility of microbiological contamination in the further concentration process.

A combination of concentration and thermal gradient through the OD membrane was obtained by using calcium chloride dehydrate 6M as a stripping solution and bulk temperatures of feed and osmotic sides of 35 and 22°C, respectively. Evaporation fluxes were in the range of 4.5-5.0 kg/m^2h for all the investigated juices. Total antioxidant activity variation of the final products coming from the UF-OMD sequence confirmed the assumptions of a mild fruit juice concentration method.

Bèlafi-Bakó and Koroknai [46] evaluated the performance of MD, OD and OMD in the concentration of apple juice. In similar operating conditions (temperature difference 15 °C, 6 M $CaCl_2$) driving forces were accumulated in the coupled operation producing the highest flux.

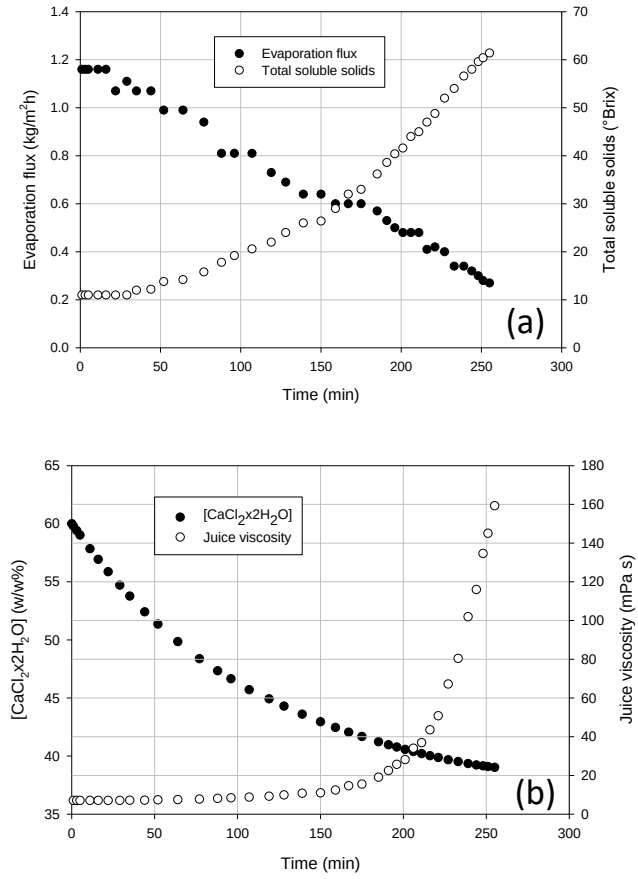

Fig. 6. Concentration of clarified cactus pear juice by OD. Time course of: (a) evaporation flux and total soluble solids; (b) brine concentration and juice viscosity (adapted from [49]).

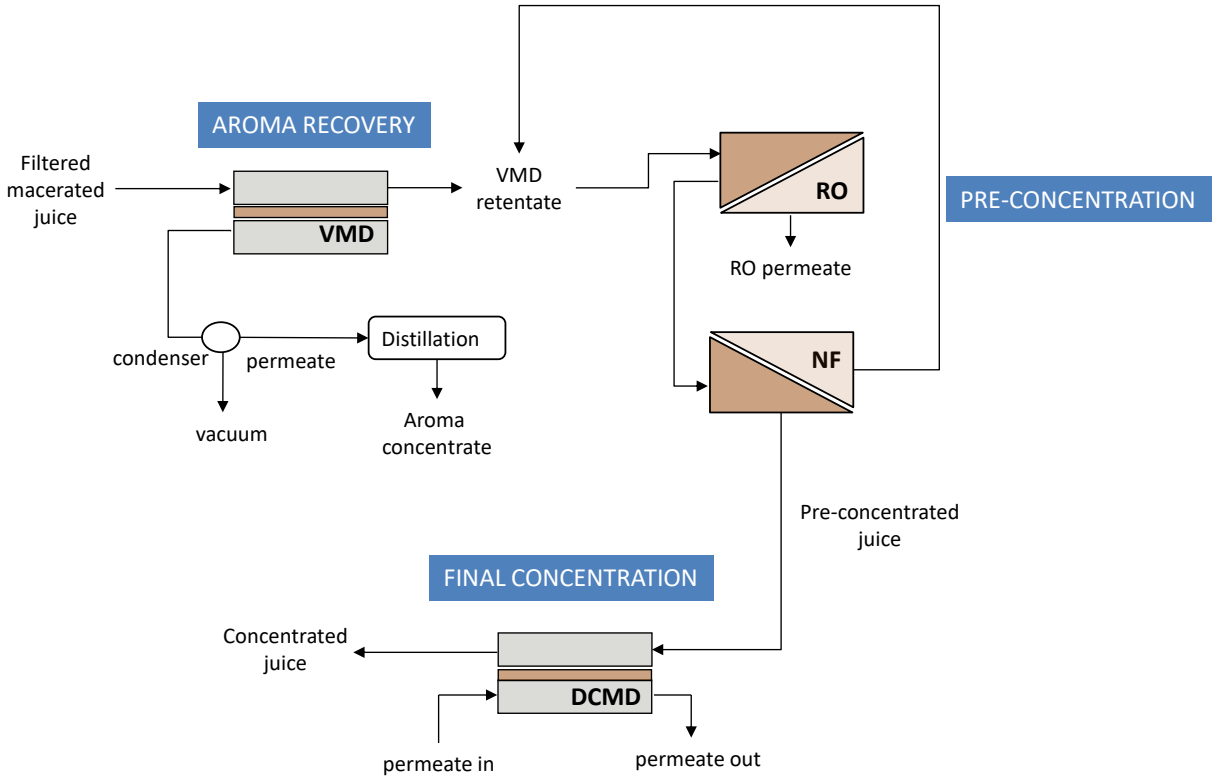

Fig. 7. Process layout for aroma recovery and juice concentration based on membrane processes (adapted from [85]).

Similarly, an increase of evaporation flux of about 20% was observed by Laganà et al. [38] in the concentration of apple juice with hollow fiber PP membranes when the mole fraction of $CaCl_2$ used as osmotic agent in combination with DCMD was increased up to 0.35.

Clarified cherry fruit juice with a TSS content of 12.6 °Brix was concentrated by OMD up to 51.45 °Brix with average evaporation fluxes of about 5.33 kg/m^{2}h and high retention of valuable compounds [86].

Onsekizoglu et al. [54] evaluated the quality of apple juice concentrated by thermal evaporation, OD, MD and OMD after a clarification step with UF membranes. Phenolic compounds, organic acids and sugars resulted very stable against all concentration processes, including thermal evaporation. Thermally evaporated samples showed the most significant color loss in comparison with clarified juice; on the other hand, the colors of samples concentrated by membrane-based processes were almost completely preserved, indicating the absence of Maillard reactions, as confirmed by subsequent analysis of 5-hydroxymethylfurfural. The OMD operation reduced trans-2-hexenal losses drastically tending towards that of the initial juice resulting as the most promising alternative to conventional thermal evaporation technique.

The coupled operation of MD and OD resulted also promising for the concentration of clarified pomegranate juice, allowing higher concentrations to be reached in shorter periods of operation with a slight increase (10 °C) in temperature of the juice [61]. Initial evaporation fluxes in the concentration of tomato juice by OMD resulted also higher (1.97 kg/m^{2}h) than those observed in OD (1.07 kg/m^{2}h) and in MD (0.94 kg/m^{2}h) [87].

Recently, OD and DCMD were investigated individually, as well as in a combined process, for the concentration of anthocyanins from aqueous extracts of muscadine grape pomace [88]. The extracts were concentrated by using ethylene chlorotrifluoroethylene (ECTFE) and PP flat-sheet membranes with a nominal pore size of 0.2 µm (from 3M, USA). The OD was performed at 22 °C using NaCl 4M as osmotic agent. For DCMD experiments, given the temperature sensitivity of anthocyanins, the feed was maintained at 40 °C and the permeate (deionized water) at 10 °C. In combined OD-DCMD experiments a similar thermal gradient was applied recirculating NaCl on the permeate side. Evaporation fluxes obtained for the PP and ECTFE membranes were similar in the OD process although at higher imposed driving forces, the PP membrane showed higher fluxes. The combination of OD and DCMD produced the highest flux with an initial value of 16.7 L/m^{2}h for the PP membrane and 15.6 L/m^{2}h for the ECTFE membrane (Figure 8).

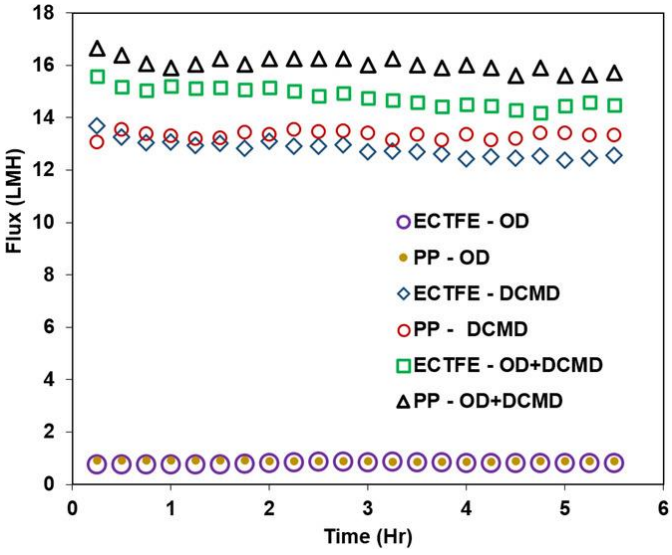

Fig. 8. Concentration of anthocyanins from muscadine pomace extract by OD, DCMD and OD-DCMD. Evaporation flux as a function of time [88].

3.2. Milk and dairy

Dehydration processes for the production of milk powder require significant amounts of energy (around 11 MJ/kg powder) most of which (96%) is consumed in evaporation and spray drying processes [89]. MD is a valid alternative to reduce the energy consumption of conventional multiple-stage falling film evaporators. Mild operating conditions used in MD are favorable to diminish protein denaturation. In addition, MD is able to use low grade heat obtained from waste heat of other processes or solar heat [90].

Whey protein concentrates are produced by concentrating the whey content from 6.5% (w/w) dissolved solids (DS) to 20% DS by UF followed by diafiltration to remove dissolved salts and sugars. An 80% DS whey

protein product is then produced through a combination of thermal evaporation and spray drying [91]. The use of DCMD as an alternative to thermal evaporation was investigated by Christensen et al. [92]. A whey protein concentrate of at least 25% protein was obtained by using a monotubular PP membrane module (Microdyn-Nadir Gmbh, Germany) fed with whey inside the tube and cold strip water on the shell side. The optimal temperature for the whey protein concentrate was 55°C leading to a much gentler process than evaporation. DCMD produced a high quality of whey concentrate minimising protein denaturation; however, low fluxes were meaured at higher concentrations (around 5.2 mL/m^{2}min, at 29% DS) due to severe concentration and temperature polarisation phenomena. Thinner membranes and higher flowrate were suggested for industrial application. Thermal and chemical pretreatment methods combined with optimized operating parameters have been also suggested to reduce the intensity of fouling in the concentration of whey by DCMD [93].

Polytetrafluorethylene (PTFE) flat-sheet membranes of 0.5 µm nominal pore-size (from Ningbo Changqui Porous Membrane Technology, China) were used for concentrating whole milk, skim milk and whey by DCMD keeping the warm feed/retentate and cold permeate stream temperatures of 54 °C and 5 °C, respectively [94]. Evaporation fluxes were found to decrease with an increase of dry-matter concentration in the feed. Retention of dissolved solids was found to be close to 100% and independent of the dry-matter concentration in the feed. Fouling phenomena resulted time-dependent for whey solutions, while for skim milk were mainly attributed to dry-matter concentration.

Cross-flow velocity was found to influence performance during skim milk processing but not during whey processing. At higher cross-flow velocities (about 0.141 m/s), fluxes were comparable to those found with reverse osmosis (12 kg/m^{2}h and 20 kg/m^{2}h for skim milk and whey, respectively, at 20% dry matter concentration). Lower feed and higher permeate temperature was found to reduce fouling in the processing of both dairy solutions [95].

Analyses of membrane fouling revealed that it is primarily driven by a combination of proteins and calcium. On the other hand, lactose did not seem to interact with the membrane polymer directly but it can be deposited once an anchor point to the membrane is established by other components. Caseins of skim milk showed strong adhesion to the polymeric membrane preventing interactions of other components; however salts were needed to form a thick and dense cake layer. Whey proteins had a weaker attractive interaction with the membrane and adhesion depended more on the presence of phosphorus near the membrane surface [96]. In addition, whey components, including minerals and proteinaceous material, were found to penetrate into the membrane matrix while skim milk caseins seemed to form a protective layer on the membrane surface [97].

Kujawa et al. [98] compared the transport properties and fouling phenomena of polypropylene (PP) and polytetrafuoroethylene (PTFE) membranes employed in the concentration of whey and lactose solutions by AGMD. The fouling propensity of the PTFE membrane was attributed to a higher contribution of the polar part (29%) of the surface free energy (SFE); on the other hand, the lowest value of normalized flux decline observed for the PP membrane was attributed to the smallest contribution of polar interaction in SFE, the lowest value of roughness (RSM), contact angle (CA), and contact angle hysteresis (HCA) evaluated after membrane utilization.

Recently, Gül and Hasanoğlu [99] investigated the effect of brine concentration, temperature, feed and brine flowrates on evaporation fluxes obtained in the concentration of milk by OD. An integrated process OD/DCMD was also studied in order to avoid dilution of the draw solution in OD. Evaporation fluxes obtained in the hybrid process resulted higher than those obtained in OD.

An optimal process design to concentrate milk from 9% to 50% solids has been recently proposed by Moejes et al. [100]. In this approach milk is concentrated by a two-stage RO section to the upper boundary of 18% solids and then up to 50% solids through a single-stage AGMD section (Figure 9). AGMD has the advantage of internal heat recovery when compared to DCMD. Nevertheless, the energy costs are quite high due to the high product recirculation to achieve sufficient cross flow along the membrane.

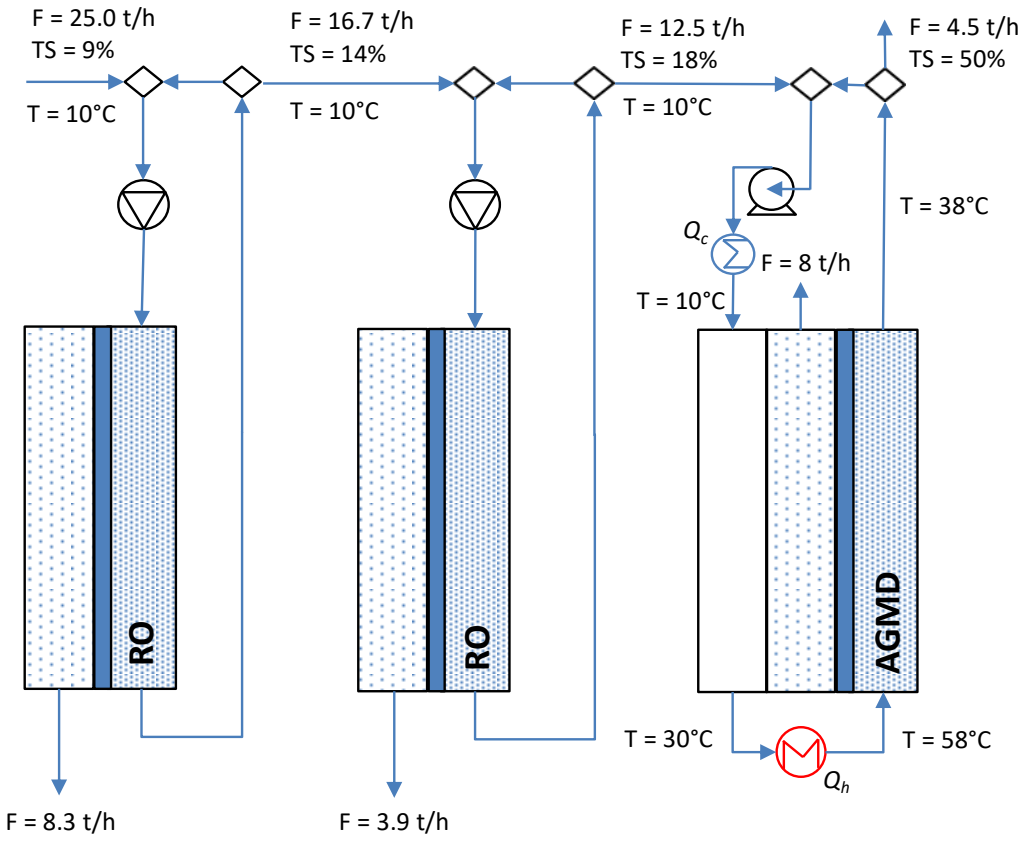

Fig. 9. Milk concentration through a combination of RO and AGMD systems [100].

3.3. Wine processing

Wine is considered the most popular alcoholic drink in the world, of high commercial importance, representing a significant pillar for the food industry with relevant contribution in terms of employment and revenues [101]. The winemaking process includes several unit operations (pressing, decanting, filtration, bottling) and processes (alcoholic and malolactic fermentations) that convert grapes into wine. This means that the quality of wine is strictly related to the composition and variety of grape.

Wine is a very complex mixture of different compounds, many of them present at very low concentrations; however, they play an important role in its evolution and quality. The components of crude wine include water, ethanol, glycerol, polysaccharides, microorganisms, different types of acids, phenolic compounds, volatile compounds, yeasts, and large particles as potassium hydrogen tartrate [102]. Among the different parameters that influence the quality of wine, the alcohol concentration is important for aging, stability and organoleptic properties of wine. The natural alcohol of wine can be increased in several ways including the treatment of grape must with additive (i.e. addition of sugars or ethanol) or subtractive techniques (i.e. reduction of water content). The addition of must concentrate (MC) or rectified must concentrate (RMC) is a typical practice to increase the sugar content of grape must; however, these methods could affect the quality of wine due to the presence of several non-sugar compounds (e.g. polyphenols, organic acids and salts) as in MC or a dilution effect in RMC.

Membrane processes such as MD and OD represent a valid alternative for grape juice concentration if compared with traditional methodologies [75]. These processes allow to increase the amount of sugars thus improving the quality of wine obtained after fermentation without addition of non grape components that can increase the wine volumes and modify its organoleptic characteristics. The possibility to operate at room temperature, preserving the quality of wine, the absence of caramelization reactions and the maintenance of sensorial and nutritional properties of the product are additional advantages.

Versari et al. [103] investigated the concentration of white, rosé and red grape juice on pilot scale equipped with plate and frame modules. The OD process significantly improved sensory quality of red wine that was judged as having a full body, more structure, and persistence.

Rektor et al. [104] compared MD and OD processes during the concentration of pre-treated grape juice by using a PP hollow fiber membrane module (020CP 2N, from Microdyn) with an effective surface area of 0.1 m^2 and pore size of 0.2 μm. Both membrane processes allowed to reach high concentration levels (up to 60 ° Brix for MD) without affecting the quality of grape juice.

Similar results were obtained by Kujawski et al. [105] in the concentration of red grape juice by OD with flat-sheet PTFE membranes of different pore size (0.2, 0.45 and 1.2 μm, from Sartorius). Experimental results showed that the membrane pores size did not affect the performance of the process in terms of evaporation flux. The total polyphenols content and the antioxidant activity of the juice were very well preserved during the OD process.

An integrated process based on the use of RO and VMD was considered as the best configuration for the concentration of grape must [106]. The RO step was designed for the concentration of must from 20 to 30 °Brix, while the VMD step was designed for the residual concentration up to 50 °Brix through Accurel V8/2 membranes, at 60 °C and 30 mbar.

Over the last years, the demand of beverages with low or zero alcohol content is fast growing for religious or healthy reasons and more restrictive policies in alcohol consumption. These considerations have focused wine industry on reducing alcohol concentration in wines, ideally without compromising wine flavour, consumer acceptance or increasing the cost of production. The techniques for reducing alcohol in wines include pre-fermentation, viticultural and microbiological strategies (i.e. development of yeast inocula that reduce the efficiency of ethanol production), post-fermentation practices (i.e. blending of high and low alcohol wine) and membrane processes [107-109]. Among them, the OD has been introduced as a promising technology for obtaining low alcohol wine with minimal changes to the sensorial properties of the product [110].

One of the first applications of OD in wine dealcoholization was investigated by Hogan et al. [2]. In this approach the alcohol content of wine was reduced up to 6% with minimum loss of its flavor and fragrance components at a temperature of 10-20°C using plain water as stripping liquid.

Varavuth et al. [111] investigated the potential of wine dealcoholization by OD by using three different types of stripping solution (pure water, glycerol 50% (w/w) and $CaCl_2$ 40% (w/w)) and a membrane contactor unit equipped with PVDF hollow fiber membranes with pore size of 0.2 μm. Experimental results indicated that ethanol removal and ethanol permeate flux increased by increasing feed and stripping velocity as well as the operating temperature. In addition, the use of water as stripper produced higher ethanol flux and lower counter transport of water due to the water activity differences when compared to other stripping solutions. The ethanol concentration of the wine was reduced up to 34% of the initial concentration after 360 min of continuous operation.

Liguori et al. [112] evaluated the effect of process parameters on partial dealcoholization of *Aglianico* red wine (12.5 %vol) and model hydroalcoholic solutions by using a Liqui-Cel micromodule 0.5 × 1 containing PP hollow fiber membranes and distilled water as stripper. The ethanol flux increased by increasing the stripping flowrate in both turbulent and laminar flow regimes. However, when the stripping agent flowed under transition or turbulent regime (Re > 2,000) a lower dealcoholization efficiency of the membrane was found. The dealcoholization rate increased by increasing the feed flowrate up to 1.2 mL/min; further increases in flowrate (3.6-5.6 mL/min) did not produce a significant effect. Therefore, the optimal conditions for ethanol removal from model solutions were obtained working in laminar conditions for both feed and stripping stream. The increase in vapour pressure difference across the membrane with an increase of ethanol concentration in the feed solution (from 10 to 15 %vol), resulted in a decrease of ethanol flux through the membrane, probably due to saturation phenomena. On the other hand, an increase of the feed temperature (in the range 15-35 °C) improved the ethanol flux according to the higher vapour pressure of ethanol at higher temperatures.

The alcohol content of red wine was reduced of 15% at an operating temperature of 20 °C without affecting its chemical and physical parameters including total volatile acidity, colour intensity, total polyphenols and organic acids content. However, the color intensity and tonality of wine samples changed when the alcohol reduction was higher than 6.5 vol.% [113].

A two-stage membrane process based on the use of RO and OD for wine dealcoholization has been patented by the Australian enterprise Memstar [114]. In this process the alcohol rich permeate from RO is recirculated through the membrane contactor together with a counter-flow of water as a stripper (Figure 10). Alcohol passes through the membrane from the permeate into the water. The dealcoholized permeate is then cooled and recombined with the wine, lowering the alcohol of the blend.

The combination of RO and OD in a sequential design for wine dealcoholization has been also recently investigated by Russo et al. [115]. Red wine (cv. *Montepulciano d'Abruzzo*), with an initial alcohol of 13.2 %v/v, was first treated by RO and then by OD up to reduce the alcohol strength of about 5, 6 and 8 %v/v. The combined RO/OD process resulted in a better preservation of the main chemical properties (i.e. total acidity, tartaric acid, pH, volatile acidity, colour intensity, total anthocyanins and total polyphenols) and volatile compounds of the dealcoholized wine samples in comparison to samples obtained from simple OD.

3.4. Olive mill wastewaters (OMWs)

The olive oil production, an agro-industrial activity of many Mediterranean countries, is associated with the generation of large amounts of dark liquid effluents called olive mill wastewaters (OMWs) with high electric conductivity, composed of 83.4% of water, 1.8% of inorganic salts and 14.8% of organic compounds on average. The pH of OMWs is in the range 4.9-5.3 due to the presence of organic acids such as malonic, citric, tartaric, lactic, fumaric, ossalic and succinic. The organic fraction, contributing to the high polluting load of these wastewaters, contains tannins, polyphenols, polyalcohols, pectin, sugar, lipids, proteins and organic acids [116]. Recently, in relation to the major interest of natural substances with biological activities, researches have been also oriented to the recovery and concentration of polyphenols as high added value compounds, transforming OMWs from effluents to raw material with high potential economic value [117]. Indeed, these compounds are characterized by a high spectrum of biological activity, including antioxidant, anti-inflammatory, antibacterial and are of great interest for the cosmetic and pharmaceutical industries and in food processing [118]. Membrane processes represent promising and advancing technologies for the recovery of water and polyphenols from OMWs. These processes, mostly in a sequential form or combined between them or with other separation technologies, have been successfully used to achieve high levels of purification and concentration of OMWs [119]. In particular, MF, UF are used as pre-treatment processes, while NF and RO are used for fractionation and final concentration of target compounds.

More recently, OD and MD have emerged as new technologies with great potential in OMWs treatment [120]. These processes allow to reach higher concentration of dissolved solids when compared with conventional pressure-driven membrane operations, and to reduce the operating costs due to low pressures applied and low membrane fouling.

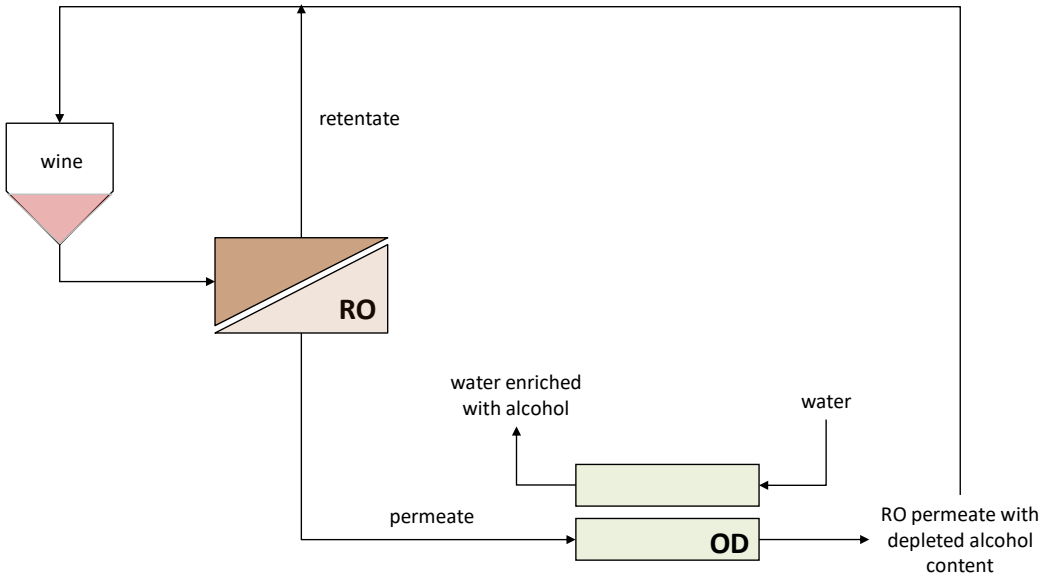

Fig. 10. Integrated process RO/OD for wine dealcoholization [114].

In the work investigated by Garcia Castello et al. [121], MF and NF processes were combined with OD and VMD in order to recover and concentrate polyphenols from OMWs. Raw wastewaters were pre-treated by a tubular ceramic MF membrane in Al_2O_3 with a pore size of 200 nm. This step allowed to reduce suspended solids and total organic carbon (TOC) of 91% and 26%, respectively, while about 78% of the initial content of polyphenols was recovered in the MF permeate. This fraction was then processed by a PES NF membrane in spiral-wound configuration (N30F, from Microdyn Nadir): the TOC of the permeate fraction was reduced of about 62% (up to 5.6 g/L, in comparison to the MF permeate) while the content of polyphenols was almost preserved. The NF permeate was recirculated in the shell side of the OD membrane module (Liqui-Cel Extra-Flow 2.5x 8', Hoechst Celanese) while calcium chloride dehydrate solution at 60 w/w% used as a stripping solution was recirculated in the lumen of hollow fibers. A concentrated solution containing 0.5 g/L of polyphenols, with hydroxytyrosol representing 56% of the total phenolic content, was obtained at a volume reduction factor (VRF) of 3. The initial evaporation flux of about 1 kg/m^{2}h decreased up to 0.35 kg/m^{2}h due to the dilution of the stripping solution and a consequent reduction of the driving force of the process (Figure 11). Steady-state evaporation fluxes of about 8 kg/m^{2}h were obtained in the treatment of the NF permeate by VMD with the use of a PVDF membrane with a pore size of 0.2 µm. However, in terms of process efficiency, the energy consumption of VMD was higher than OD due to the use of a vacuum pump and a refrigerator step to condense the permeate.

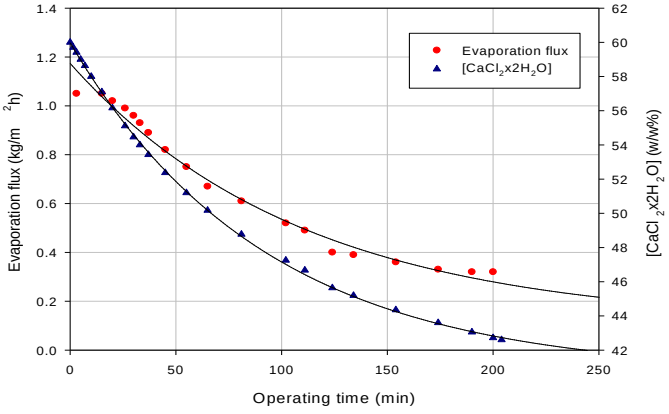

Fig. 11. Osmotic distillation of nanolfiltered olive mill wastewaters (NF retentate). Time evolution of permeate flux and concentration of osmotic agent (adapted from [121]).

In another approach OD was used to produce an enriched fraction of phenolic compounds from a NF retentate after an acidification/MF step aimed at removing suspended solids from the raw wastewater [122]. In the OD process, performed with a Liquicel Extra-Flow 2.5×8″ membrane contactor, the NF retentate was recirculated in the shell side while calcium chloride dihydrate at 60 w/w% was recirculated counter currently in the tube side. The concentration low molecular weight phenolic compounds in the final product was in agreement with the concentration factor of the process (~7). The concentrated polyphenolic stream was finally encapsulated in a water-in-oil emulsion by membrane emulsification. According to the estimated mass balance of the process, 1.43 kg of phenolic compounds (85% of the initial phenolic content) and 800 L of purified water (80% of the initial volume) can be obtained from 1 m^3 of raw wastewaters (Figure 12).

The concentration of polyphenols from OMWs by DCMD was investigated by El-Abbassi et al. [123] with the use of two microporous hydrophobic membranes (TF 200, in PTFE, from Gelman and GVHP, in PVDF, from Millipore). Permeate fluxes obtained with the TF 200 membrane resulted higher (of about 7 L/m^{2}h at a ΔT of 20 °C) than those obtained with the GVHP membrane having greater thickness. The TF 200 membrane exhibited also a better separation coefficient (99%) and a higher concentration factor (1.72). The evaporation fluxes measured for the TF 200 membrane with microfiltered wastewaters resulted also higher than those measured with crude OMWs and wastewaters pretreated by coagulation/flocculation process [124].

Authors compared also the performance of three microporous PTFE membranes with a pore size of 0.2, 0.5 and 1.0 µm (TF 200, TF 450 and TF 1000, respectively, all from Gelman) in the concentration of polyphenols from OMWs by DCMD [125]. Results indicated that the evaporation flux increased with the increase of the membrane pore size, the mean temperature and the temperature difference but no significant effect was detected between the pore size and the polyphenol separation coefficient which was of about 100%. In particular, for the TF 1000 membrane evaporation fluxes of about 27 L/m^{2}h were reached at a ΔT of 50 °C (with a feed temperature of 70 °C and a permeate temperature of 20 °C). In similar operating conditions higher evaporation fluxes were measured with selected membranes in the treatment of table olive wastewaters [126].

The same membranes were also used in OD and OMD tests performed on crude OMWs by using calcium chloride as an osmotic agent. Permeate fluxes were of the order of 2.9-4.2 L/m^{2}h and the obtained concentration factor of phenolic compounds was up to 1.2 after 280 min of crude OMW processing by OD. When using OMD with a temperature difference of 20 °C and a mean temperature of 30 °C, this concentration factor reached a value up to 1.9 after 30 h of operating time using the membrane TF200 [127]. All selected membranes exhibited a high fouling resistance since the reduction of the water evaporation flux after the treatment of OMWs was lower than 5%.

More recently, Carnevale et al. [128] investigated the potential of DCMD and VMD in the treatment of OMWs by using PP capillary membranes with average pore size of 0.2 µm and inner diameter of 1.8 mm (from Membrana).

Rejections of about 99.9% towards polyphenols were measured at feed temperatures of 30, 40 and 50 °C (while maintaining the cold side at 15 °C). Evaporation fluxes at 50 °C were of about 6.5 kg/m^{2}h. An increase of permeate flux was achieved in VMD tests. In particular, at 50 °C the permeate flux was around 19 kg/m^{2}h. Rejection values increased from 97.1% to 99.6% when the feed concentration increased from 150 ppm to 2500 ppm.

4. Conclusions

Membrane distillation and osmotic distillation have proven to be attractive alternatives to thermal evaporation and low-temperature separation techniques such as reverse osmosis, ultrafiltration and vacuum freeze-drying in the processing of agro-food products and by-products. Typical applications in the concentration of liquid foods, including fruit and vegetable juices, milk, whey and grape must, as well as in wine dealcoholization, have been reviewed in the light of current literature data. Significant advantages in terms of improved product quality, maximum achievable concentration, low fouling index and low energy consumption have been clearly demonstrated with respect to standard concentration methods.

Low evaporation fluxes in MD and OD seem to be the main drawbacks, when compared with reverse osmosis and thermal evaporation, for industrial implementation. However, significant improvements of the process efficiency and economy can be achieved through the combination of MD and OD with pressure-driven membrane operations, as clearly demonstrated by several applications investigated on both laboratory and semi-industrial scale. In addition, developments of tailor-made membranes are crucial for improving the performance of non-thermal membrane processes especially in terms of water removal rate. In this view, efforts should be devoted to the development of innovative membranes with high water permeability, reduced tendency for concentration polarization, high selectivity and mechanical stability for long-term applications. The utilization of low-grade waste and/or alternative energy sources for MD and the management of the spent osmotic agents or their reinforcement during OD processes are other research areas to be explored in order to minimize both industrial waste and energy consumption and to allow large scale applications.

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