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Membrane distillation assisting the food production processes of thermally sensitive food liquid items: A review

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Membrane distillation assisting the food production processes of thermally sensitive food liquid items: A review

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Abstract

Physical separation technologies have become important tool for processing in the current food manufacturing industries, especially for the products containing bioactive compounds thanks to their health benefits in costumers. As for the processing of bioactive food ingredients implies the implementation of integrated systems oriented to their separation, fractionation, and recovery. In this field, membrane distillation (MD), which is a thermally driven membrane process, has been proposed as an alternative for the separation and concentration of liquid food items. In principle, MD can separate water and volatile compounds from aqueous feed solutions through a permeate that passes across microporous hydrophobic membranes. The separation via MD is thanks to the vapor pressure difference on both membrane sides. In this review, we analyzed the ongoing experimental efforts aimed to recover and purify food bioactive compounds from the concentration of fruit juices and extracts using MD. Also, the processing of dairy products, concentration of food by-products, and ethanol production and its removal from beverages using MD have been reviewed. Additionally, a feedback on the distinct

membrane module configurations and membrane requirements for successful operation is addressed.

Keywords: Membrane distillation (MD); thermal driven process; bioactive compounds; foods; concentration.

1. Introduction

Today, separation processes are a core part of agro-food industries since most of the bioactive substances in nature are tough to obtain in pure form. To some extent, any separation method is needed before either substance consumption or processing, this is more relevant for functional molecules and nutraceuticals. Importantly, the proposed method for the processing of food bioactive components must provide outstanding yield while minimizing the loss and bioactivity. Within the category of bioactive substances, we can find mostly phenolic compounds (including anthocyanins, phenolic acids, catechins, flavonones, flavonols, non-flavonoid polyphenol) carotenoids, oleoresins (capsaicins), polysaccharides, peptides, terpenoids, phytosterols, among many others (see **Figure 1**). The main concern, which scientists need to face during the processing of bioactive compounds, relies on their separation from complex matrix source, and eventually their polishing (purification). As a general overview, **Figure 1** illustrates a guide of the most typical unit operation required for the pre-treatment, extraction, purification and concentration stages of bioactive substances contained in natural sources. Unquestionably, the sequence of separation methods must be raised according to the type of target analyte and its origin (R. Castro-Muñoz et al., 2022). Additionally, the selected separation strategy should profit on specific physicochemical property of the substance, such as size, structure, polarity, etc., which should differ from other molecules present in the source.

In theory, each selected separation or purification technique should effectively perform the extraction at a low cost, optimal yield minimal loss of compounds (Roberto Castro-Muñoz et al., 2022; Valencia-Arredondo et al., 2020). Once extracted, the concentration of bioactive-containing aqueous solutions represents one of the most seek unit operations. In this field, particular food products require concentration technologies, such as beverages, fruit juices, vegetable and herbal extracts, milk, whey, etc. Herein, the volume reduction of concentrates through dewatering critically reduces the substances and water transport, storage and packaging cost and consequently makes them more resistant to chemical and microbial degradation.

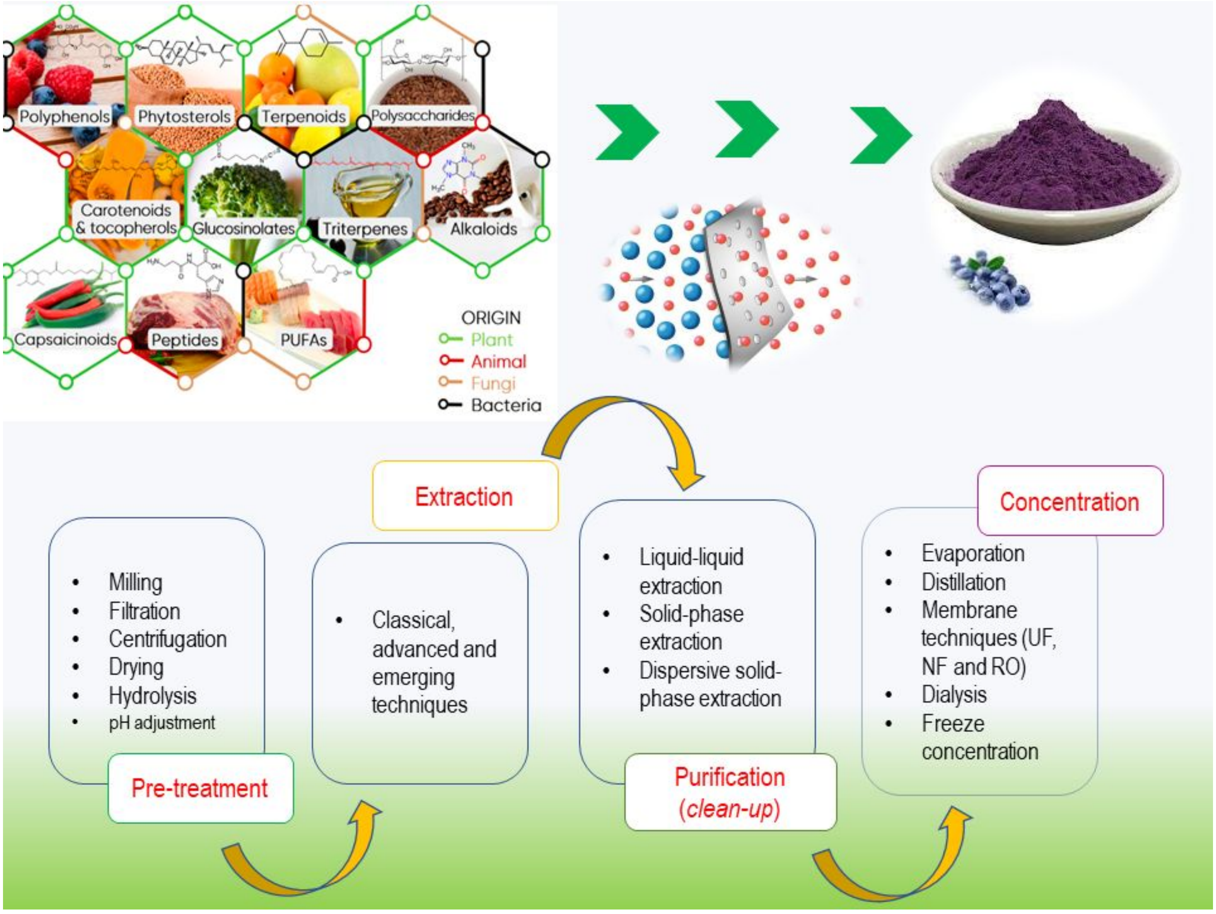


Figure 1. Main bioactive compounds extracted from natural sources and typical extraction strategy required for their concentration.

To date, typical methods employed for liquid concentration in the food industry, such as multistage vacuum evaporation, require high energy expenditure, while drastically affecting the organoleptic and nutritional attributes of the final product due to the high temperature (Charcosset, 2021; Echavarría et al., 2011). Over the last decades, many industries have conceived the raid of membrane technology in particular processes aimed at the separation of food ingredients. Membrane-based processes introduce multiple advantages during the processing of natural sources and extraction of bioactive ingredients, such as alternative for high-energy consuming evaporation process, wastewater and by-products reuse, acceptable management of wastes, and relatively low capital investments (Zhong et al., 2021). On the other hand, issues with membrane durability and maintenance, operating life, replacement costs, chemical stability, pH sensitivities and fouling issues are among the main drawbacks of membrane technologies (Julian et al., 2021). In the field of food processing, membrane processes, such as microfiltration, ultrafiltration, nanofiltration, forward osmosis, reverse osmosis and pervaporation have been proposed (Alvarez et al., 1997; Roberto Castro-Muñoz, Boczkaj, et al., 2020; Sant'Anna et al., 2012). Additionally, membrane distillation (MD), which is defined as thermally driven membrane process, has been ultimately applied for concentration of foods and natural extracts with interest due to their nutritional value. For instance, MD operates with lower temperatures and pressures compared with classic distillation process and even pressure driven membrane unit operations. Furthermore, when treating complex solutions, the implemented membranes are less prone to fouling comparing to micro and ultrafiltration, and reverse osmosis (Lawson & Lloyd, 1996; Onsekizoglu, 2012).

With the aim of giving a compelling outlook in this field, this review focuses on covering the most relevant application of MD in the food industry in terms of clarification and concentration of juices and natural extracts. Concurrently, feedback on theoretical principles and parameters influencing this process is also given, along with the main process parameters involved in the production of such food items. Apart from this, we present some advantages and disadvantages of MD technology compared with other membrane processes.

2. Thermal-driven membrane distillation: Principles, process variables and membrane requirements

Membrane distillation (MD) uses a vapour pressure gradient as the driving force, which is created between feed and permeate streams by inducing a temperature difference across the permeable and selective physical barrier (i.e. membrane). Both streams are in direct contact with the membrane that may presents a microporous structure. Typically, the membrane must own a hydrophobic character, which will fundamentally prevent the transport of the feed bulk in liquid state. At each membrane pore, liquid/gas interface is generated, in which the vapour phase permeates through the membrane and later condensate at the permeate stream.

The mass and heat transfer concurrently takes place. In theory, two relevant heat transfer mechanisms use to appear. For instance, the conductive heat transfer inside the membrane pores takes place along with the vapor diffusion, resulting in a temperature change at both membrane boundary layers. This latter phenomenon produces a temperature gradient between the feed and permeate sides and hence origins a convective heat transfer. **Figure 2** illustrates the heat flux in direct contact MD process.

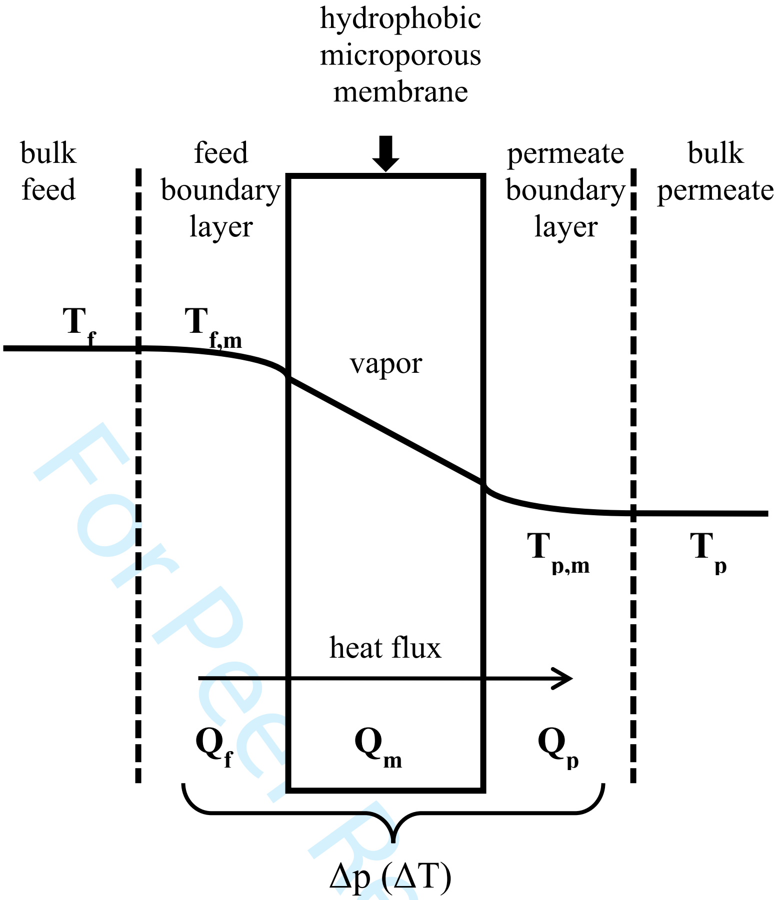


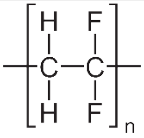
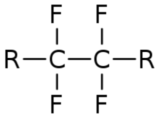
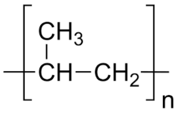
Figure 2. Graphical representation of heat flux and temperature polarization in DCMD.

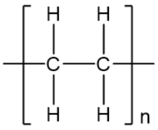
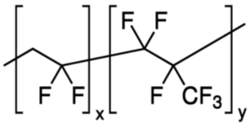
2.1. Membrane requirements

As for the membrane required for MD technology, it should own a highly hydrophobic surface, which will favorably repel water molecules in liquid phase while facilitating the permeation of the vapor. MD membranes based on polymers are generally tailored by several fabrication protocols, such as stretching, sintering or phase inversion (Curcio & Drioli, 2005). Critical membrane properties, including pore size, hydrophobicity, porosity, thickness, thermal conductivity and tortuosity, determine the separation performance of the membrane. Today's efforts are oriented to develop membranes with superior fluxes and rejections rates, and compelling features to face critical aspects that compromise the membrane performance such as the wetting phenomenon and long-term operation stability (Perrotta et al., 2017; Tijing et al., 2016; H. Zhang et al., 2018). Here, we describe some issues and desired properties in membranes for MD technology.

- *Wetting resistance:* Knowing that the membrane is in contact with liquid aqueous feed solution, the membranes must display a strong hydrophobicity for preventing the wetting while maintaining the retention of non-volatile substances. Experimentally, improved membrane surface hydrophobicity has been noted when increasing the surface roughness and lowering solid/liquid interface energy. To some extent, the membrane susceptibility to wetting phenomenon is assayed via the liquid entry pressure (LEP) parameter, which is related to the pressure needed for the liquid to permeate across the membrane. To reach high LEP value together with good wetting prevention, the desired membrane should preferentially possess, high surface tension, low energy between liquid-membrane interface and narrow (small) pore size. **Table 1** reports some commercial hydrophobic polymers used in the manufacture of MD membranes with suitable properties for such a process.

Table 1. Hydrophobic polymer membranes with relevant properties considered for wetting resistance.

Hydrophobic polymers:	**Chemical structure:**	**Water contact angle:**	**Surface energy (x 10^{-3} Nm^{-1})**	**LEP**	**Reference:**
PVDF	 $-[CH_2-CF_2]_n-$	113°	30.3	21.3 kPa	(J. Zhang et al., 2010)
PTFE	 $R-CF_2-CF_2-R$	126°	9.1	24 kPa	(J. Zhang et al., 2010)
PP	 $-[CH(CH_3)-CH_2]_n-$	116-120°	30	6-28 kPa	(K. He et al., 2011)

PE		83-108°	28-33	0.5-0.7 bar	(Zuo et al., 2016)
PVDF-HFP		125°	-	19.1 psi	(Lalia et al., 2013)

- *Membrane thickness:* This property affects the transport overall MD process in an inversely proportional way. For example, thinner membranes reduce the mass transfer resistance and thus increase the vapor flux. On the contrary, the membrane thickness affects the conductive heat loss over the MD operation. In this way, the implemented membrane should preferentially be as thick as possible to mitigate the heat loss. Unfortunately, this latter point compromises the high vapour flux requirement; therefore, the thickness needs to be optimized, e.g., it has been documented that the optimal membrane thickness for a MD process should be ranged from 30 to 60 µm (Laganà et al., 2000).
- *Membrane porosity*: This parameter is determined as a relationship between volume of the pores and the total membrane volume. Importantly, the membrane porosity is directly proportional to the evaporation surface area, thus, higher porosity of the membrane results into higher vapor flux (Susanto, 2011). In addition to this, a membrane porosity increase substantially reduces the conductive heat loss, as the conductive heat transfer coefficient of the gases entrapped in the membrane pores is typically an order of magnitude smaller than the conductive heat transfer coefficient of the hydrophobic membrane material (Lawson & Lloyd, 1996). For instance, it has been determined that the membrane porosity value for an exceptional MD must be ranged from 30 to 85% (El-Bourawi et al., 2006).

- *Membrane pore size*: As stated early, microporous structure in membranes is typically needed for MD. Also, the membrane pore size requires be optimized to prevent wetting of the membrane, and concurrently, ensure the highest flux. To some extent, the optimal pore size depends on the purpose of the application, which intrinsically depends on the kind of feed solution. Theoretically, Schneider et al. (1988) declared a pore diameter ranged from 0.5 to 0.6 μm seems to be enough to mitigate the wetting. Of course, uniform pore size over the membrane structure is desired to get a better approximation in terms of vapour flux (Susanto, 2011).
- *Pore tortuosity:* Together with the pore size, the shape of membrane pores also plays a great role in the membrane process. As a definition, the membrane tortuosity represents the pore shape deviation from the cylindrical structure. This property is given by the average length of the pores in comparison with the membrane thickness. In terms of separation performance, when the membrane pores make tortuous pathways, there is a decrement in the flux of the diffusing molecules, therefore, low tortuosity results in high vapor flux. Commonly, the most frequent tortuosity is 2 (Phattaranawik et al., 2003).
- *Thermal conductivity:* Since we seek minimizing the heat loss in MD, it is obvious to prepare membranes based on a material with low heat conductivity. The heat loss decrement results in higher energy efficiency, lower susceptibility to temperature polarization phenomena, as well as higher vapor flux. As mentioned previously, the membrane thermal conductivity is generally higher than that of gases occupying the pores. It is known that the thermal conductivities of distinct polymers, such as PP, PTFE, PVDF, are ranged from 0.11 up to 0.27 $Wm^{-1}K^{-1}$ at 23 ^{o}C (Alkhudhiri et al., 2012).

Summarizing such membrane properties, **Table 2** gives the characterization data of typical commercial membranes applied in MD. It is worth mentioning that most of these commercial membranes do not meet all the MD requirements or lacking in any of such properties. Therefore, there is a necessity of designing outperforming membranes with desirable membrane features. Apart from these latter aspects, there is also a specific drawback in hydrophobicity nature improvement of smooth membrane surfaces. This is the case of PVDF, which displays a smooth surface as a result of saturation by fluorinated methyl groups, in general, the membranes are able to achieve a maximum water contact angle of 120º (Liao et al., 2013). In this sense, a feasible pathway for improving the surface hydrophobicity is through the increase of surface roughness (Tijing et al., 2016). Apart from reaching a strong water repellence, a particular attention should be focused to design a micro- and nanostructured surface as well. Here, ongoing research has utilized nanofillers to be embedded into the polymer membrane to tailor the physicochemical properties and structure of membranes, including hydrophobicity, porosity, chemical, thermal and mechanical stability, and surface charge density. This membrane modification by filler loading is named the new generation of membranes, in which different nanomaterials have been used including carbon nanotubes (Tijing et al., 2016), graphene (Gontarek et al., 2019), clay (Prince et al., 2012), silica (Y. Zhang & Wang, 2013) and titanium dioxide (Meng et al., 2014). Depending on the nanoparticle's properties, superhydrophobic MD membranes can also be obtained.

Table 2. Properties of commercial MD membranes.

Polymer/module:	**Trade name:**	**Manufacturer:**	**Thickness (µm):**	**Mean pore size (µm):**	**Porosity (%):**
PVDF/flat sheet	GVHP	Milipore	125	0.2	80
PVDF/flat sheet	Durapore	Milipore	110	0.45	75
PTFE/flat sheet		Osmonics	175	0.22	70
PTFE/hollow fiber	POREFLON	Sumitomo Electric	550	0.8	62

PP/flat sheet	MD080CO2N	Enka Microdyn	650	0.2	70
PP/hollow fiber	Liqui-Cel® Extra-Flow 2.5×8 in	Hoechst-Celanese	50	0.044	65
PE/hollow fiber	UPE test fiber	Millipore	250	0.2	-

2.2. MD operation configurations

An important aspect playing a crucial role in the performance of MD distillation relies on the type of process configuration used. Typically, distinct configurations for this unit operation can be implemented fundamentally varying from each other in the way of vapour condensation on the permeate side, as described in **Figure 3**.

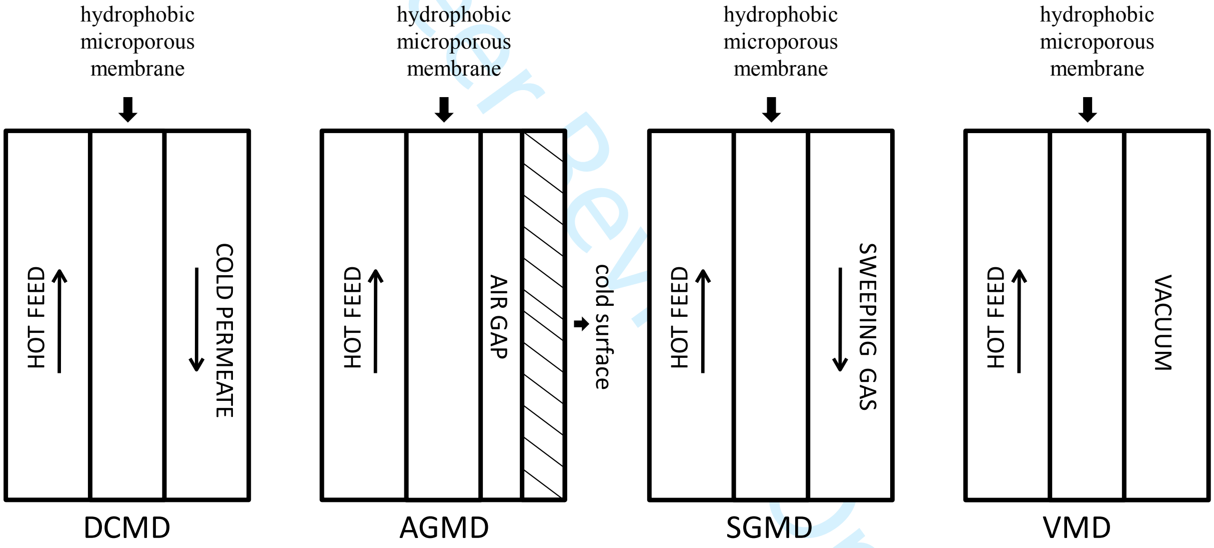


Figure 3. Different operation configuration of MD.

- Direct contact membrane distillation (DCMD): In this configuration both feed and permeate liquids are in direct contact with the hydrophobic microporous membrane. Since the permeate presents lower temperature than that of the feed, permeate is intentionally employed for the condensation of the other fluid. Thanks to its easy implementation and simplicity, this MD configuration is the most common implemented at lab scale. Unfortunately, the obvious direct contact of liquids with membrane interface results into a heat loss over the membrane. At this point, DCMD is identified as the lowest thermal efficiency among all MD operation configurations.

- Air gap membrane distillation (AGMD): In this mode, the vapour phase permeating across the membrane is condensed onto a cold surface. Herein, the membrane and cold surface are intentionally separated from each other by an air gap. In contrast to DCMD, AGMD displays less heat loss over the membrane.
- Sweeping gas membrane distillation (SGMD): Here, the vapour permeating is sweeping and carrying by using a cold inert gas outside the membrane, which is finally condensed. The low heat loss and reduced mass transfer are among the main advantages of this MD configuration, while the high operational cost compared with other configurations is identified as its main drawback making to be used rarely.
- Vacuum membrane distillation (VMD): In this configuration, the permeate stream is subjected to a vacuum that induces a driving force. Thanks to the low pressure, condensation phenomenon occurs outside of the membrane.

2.3. Types of membrane modules and requirements

To date, several membrane modules have been successfully designed for MD, such as plate and frame, spiral-wound, tubular, capillary or hollow fiber (Alkhudhiri et al., 2012). A typical membrane module aimed to be used in MD should display a high packing density. This latter property denotes the ratio of membrane area to the packing volume. Additionally, the membrane must offer high feed and permeate flow rates, which are commonly fed tangentially to the membrane (or also denominated as *crossflow* mode). Interestingly, the module design should potentially offer the chance of creating turbulent flow rate to give heat and mass transfer among bulk solution and the solution at boundary layer. In this sense, prevention of the temperature polarization and concentration polarization effects can be observed.

As part of required characteristic for MD membranes, the module needs to provide a low pressure drop over the module length to mitigate the flooding of membrane pores, which is generally caused by a high transmembrane hydrostatic pressure. Also, the membrane module should guarantee unchanged temperature profile of the liquids over the module length, together with minimal heat loss. Regarding the membrane material, it should be selected considering the treated feed solutions that can compromise the structural properties of the membranes, e.g., corrosion.

3. Findings on MD processes for the processing of food items

3.1. Clarification and concentration of natural extracts and fruit juices

As most of the membrane unit operations, MD can be operated with mild temperatures and atmospheric pressures; these operational conditions make it suitable for juice concentration containing thermolabile bioactive substances (Bhattacharjee et al., 2017). In other words, this process can potentially minimize the degradation of such high-added value molecules. In pioneering works, such hypothesis has been proven by Calabrò et al. (1994), who evaluated the performance of microporous PVDF membrane for the concentration of orange juice. The researchers noted exceptional retention of different molecules, such as soluble solids, sugars, and organic acids. Considering such findings, the research community supported the exploration of this membrane technology confirming it as a potential alternative aiming the concentration of distinct fruit juices, including orange (Deshmukh et al., 2011), apple (Gunko et al., 2006), black currant (Bagger-Jørgensen et al., 2004), kiwi fruit (A. Cassano & Drioli, 2007), pineapple (Hongvaleerat et al., 2008), grape juices (Rektor et al., 2006), to mention just a few. **Table 3** enlists the main applications of MD dealing with the concentration of fruit juices, applied operating conditions and unit operation configuration and the most relevant outcomes.

Table 3. Application studies of MD aimed at the concentration of fruit juices.

MD configuration	Operating conditions:	Membrane type:	Fruit juice:	Flux:	Concentration efficiency:	References
DCMD	Tf : 37 °C Tp : 28 °C	commercial plate PVDF	orange juice	~ 0.4 kg/m2/s $*10^3$	concentration up to 400 g/L	(Calabrò et al., 1994)
DCMD	Tf : 24 ± 1 °C Tp : 17 °C	hollow fiber PP	blood orange juice	0.6 kg/m^2h	65 °Brix	(Quist-Jensen et al., 2016)
DCMD	Tf : 70 °C Tp : 10 °C	flat sheet PVDF	apple juice	28 $Lm^{-2}h^{-1}$	to 60–65 °Brix	(Gunko et al., 2006)
OD	Tf : 35 °C Tp : 20 °C	flat sheet PTFE	pineapple	10 kg/m^2h	10.6 to 27.8 g 100 g-1 TSS	(Hongvaleerat et al., 2008)
DCMD	Tf : 30 °C Tp : 11 °C	hollow fiber PP	black currant	0.8 kg/m^2h	58.2 °Brix	(Kozák et al., 2009)
DCMD	Tf : 32 °C Tp : ~4 °C	hollow fiber PP	apple juice	1 kg/m^2h	64°Brix	(Laganà et al., 2000)
OMD	Tf : 35 °C Tp : 20 °C	flat sheet PTFE	cactus pear juice	3-4 $Lm^{-2}h^{-1}$	23.4 °Brix	(Terki et al., 2018)
DCMD	Tf : 30 °C Tp : -	capillary PP	grape juice	2-2.5 kg/m^2h	65 °Brix	(Rektor et al., 2006)
OD	Tf : 40 °C Tp : 40 °C	hollow fiber PP	cranberry juice	1.21 $Lm^{-2}h^{-1}$	48 °Brix	(Zambra et al., 2014)
OMD	Tf : 35 °C Tp : 20 °C	flat sheet electrospun based on PSF/zeolite	Lemon juice	-	34.5°Brix	(Estedlali et al., 2021)
DCMD	Tf : 30 °C Tp : 10 °C	Commercial hollow fiber PP	Tomato juice	0.94 $Lm^{-2}h^{-1}$	40 °Brix	Savaş Bahçeci et al. (2015)

DCMD	Tf : 35 °C Tp : 20 °C	flat sheet PVDF	Propolis extract	0.94 kg m^{-2}h^{-1}	-	(Hamzah & Leo, 2018)
DCMD	Tf : 70 °C Tp : 20 °C	flat sheet PVDF	Ginger extract	-	-	(Zou, Hu, et al., 2022)
DCMD	Tf : 70 °C Tp : 20 °C	PVDF/PSF hollow-fiber membranes	Ginger extract	-	-	(Zou, Kim, et al., 2022)
VMD	Tf : 45 °C Tp : 27 °C	Microporous PP	Coffee products	-	-	(Criscuoli & Drioli, 2019)

Experts in the field have observed that the flux in MD depend on process parameters, such as both applied temperatures, along with temperature difference generated across feed and permeate, feed concentration and both flow rates of feed and permeate (Alfredo Cassano & Drioli, 2010). In theory, high feed temperature in MD process is suggested to foster the evaporation efficiency (EE). This latter parameter is calculated as the ratio between the heat that contributes to evaporation to the total heat input in the membrane module (Smolders & Franken, 1989). Unfortunately, towards the treatment of fruit juices containing thermolabile bioactive substances, the process operation at high temperatures is not proposed since the overall quality is compromise, along with the inherent formation of undesired substances, such as hydroxymethyl furfural and furan (Crews & Castle, 2007; Vranová & Ciesarová, 2009). Furthermore, feed temperature increase fosters a higher prone to the temperature polarization effect (Hwang et al., 2011). This is one of the most important facts when aiming for the optimization of operating temperature profile.

Bagger-Jørgensen et al. (2011) evaluated two MD configurations in recovering aroma molecules from black currant. Initially, the authors investigated the influence on key operation parameters, such as feed temperature and feed flow, on the permeate flux and concentrate properties and quality. Here, twelve aroma substances were targeted for the determination of concentration factor, which in fact they were found to be the highest at the highest feed temperature. For instance, the process showed that for the volatile aromas with the most hydrophobic nature displayed concentration factors ranged from 9.3 up to 12.1 (at 45ºC). Also, the highest operating temperature of the feed and its flow rate of 400 L/h revealed an aroma recovery of approximately 84 vol.%. It is important to note that the researchers made a comparison with the recovery efficiency between SGMD and VMD. In general, SGMD process seemed to be less affected by the flow rate but was more impacted by the temperature. While the concentration by VMD configuration shortened the operating time since the higher flux was obtained. On the contrary, as the longer time demanded by SGMD for the concentration, a higher loss of bioactive substances, such as anthocyanins and polyphenols, was acquired in comparison with the VMD.

In a different work, Onsekizoglu et al. (2010) also used MD for apple juice concentration, revealing that the feed flow rate had null effect on the transmembrane flux as compared with of temperature difference effect. In the MD operation, the initial clarified apple juice presented total soluble solids (TSS) of 12 ºBrix, a value which was reached to be up to 65 ºBrix after concentration. Regarding the impact of the process on the juice quality, both nutritional and sensorial properties remained like the original juice. The authors concluded that MD, unlike conventional thermal evaporation, contributed to maintaining concentrated juice's natural color appearance and attractive aroma.

As a key principle, it is crucial to control the constant trans-membrane vapor pressure to mitigate a loss in permeate flux in MD (Laganà et al., 2000). In the line of this analysis, Quist-Jensen et al. (2016) in their work concentrated a clarified orange juice via DCMD. The researchers observed an evaporation flux decrement at the preconcentration step, which was credited by the decrease in temperature difference between the feed and permeate. Surprisingly, the outcomes declared that in such final concentration stage, the trans-membrane flux decrement can also be influenced by the juice viscosity increase. Testing two-step DCMD process, the TSS in clarified orange juice has increased from 9.5 up to 65 °Brix. Supporting Jensen´s results, Gunko et al. (2006) also experimented a significant temperature gradient dependence on the DCMD operation. The outcomes exhibited that the cooling water temperature decrease, from 30°C to 10°C, improved the flux ca. two times (180%) when operating at 50 °C. Unfortunately, the same decrease in cooling water temperature for the feed temperature equal 70°C resulted in only 10% flux improvement. In terms of the best permeate flux (ca. 28 LMH), this was observed at the beginning of the concentration process. Once the concentration initiated to be reached, there was a decrement in permeate flux, e.g., when TSS content was 50 °Brix, the permeate flux decreased until 9 $L/m^2/h$, which continued to decrease until reaching a concentration between 60–65 °Brix. Similar TSS (ca. 64 °Brix) was obtained by Laganà et al. (2000), who proposed polypropylene hollow-fiber modules for DCMD configuration aimed to concentrate apple juices. In this work, the authors noticed that flux was majorly dependent on temperature polarization coefficient, rather than concentration polarization coefficient which was found to be negligible.

An important constraint in the concentration of fruit juices using membrane technologies (like MD) relies on the membrane fouling. This latter phenomenon occurs due to the content of colloidal particles in the extract which may cause clogging or blocking of membrane pores (Pichardo-Romero et al., 2020). For instance, Mirsaeedghazi et al. (2009) eventually noted cake layer formation on the membrane surface which inevitably cased serious membrane fouling, such event occurred within the first 5 min of operation during the processing of pomegranate juice. Apart from, the membrane fouling, the layer deposited also promoted the membrane wetting and thus resulted in the transport of the liquid across the membrane. Usually, carbohydrate foulants, such as cellulose, pectins, lignin and hemicelluloses, are found in fruit juices.

Knowing the effect of membrane fouling on the process operation of MD modules, the researchers started to face membrane fouling by strategically integrating different membrane unit operations and pre-treatments. For example, He et al. (2007) clearly observed that the pre-clarification of the apple juice helped to obtained higher productivity in terms of permeate flux in UF process. Here, enzymatic pretreatment has been proposed as initial step of juice clarification by hydrolyzing the pectin and other polysaccharides (Onsekizoglu et al., 2010). After such pre-treatments and clarification using membranes, MD can be implemented obtaining more efficient and less prone to fouling processes. The preliminary filtration is typically suggested to remove suspended solids and macromolecules from the fruit juices, which consequently contributes to reduce the extract viscosity and thus acquire higher fluxes during MD concentration stage. It is known that the reduction of juice viscosity enhances hydrodynamic conditions in the membrane pores and therefore decreases the concentration and temperature polarization phenomena (Lukanin et al., 2003).

Integrating macromolecular sieving clarification (like MF) with MD, Rektor et al. (2006) clarified and concentrated grape juice, respectively, in which the final concentrate extract was subjected to a subsequent water implementing another membrane technique, such as reverse osmosis (RO). In a more recent development, Onsekizoglu (2013) combined UF and MD operations to concentrate pomegranate juice evaluating their impact on product quality and process performance. Initially, the UF stage contributed to improve the physical aspect of the juice while maintaining valuable substances such as organic acids. The ultrafiltration confirmed again the removal of macromolecular compounds, which may consequently provoke membrane wetting and can also produce a non-allowable convective flow of liquid through the post treatment with the MD membrane. Overall, integrated membrane operations permitted to produce a concentrated pomegranate juice containing 57 °Brix with acceptable content of phenolic compounds, total monomeric anthocyanins, pH and color.

Other membrane technologies, such as reverse and forward osmosis, have been utilized for the pre-concentration stages of natural extract prior MD. Several cases of study haven documented proposing such processing strategy, e.g., acerola juice (Pagani et al., 2011), blood orange (Galaverna et al., 2008), and citrus and carrot juices (A. Cassano et al., 2003). Implementing three different membrane stages, Kozák et al. (2009) successfully concentrated blackcurrant juice concentration using DCMD, however, the initial extract was firstly clarified with MF, followed by preconcentration via reverse osmosis. Both membrane processes (MF and RO) contributed to concentrate the juice from 15 to 22 ºBrix, afterwards, DCMD greatly concentrated the extract until 58 ºBrix by applying only a temperature difference of 19 °C. Similar to Kozak´s study, Sotoft et al. (2012) also concentrated blackcurrant juice integrating various membrane operations, as described in **Figure 4**.

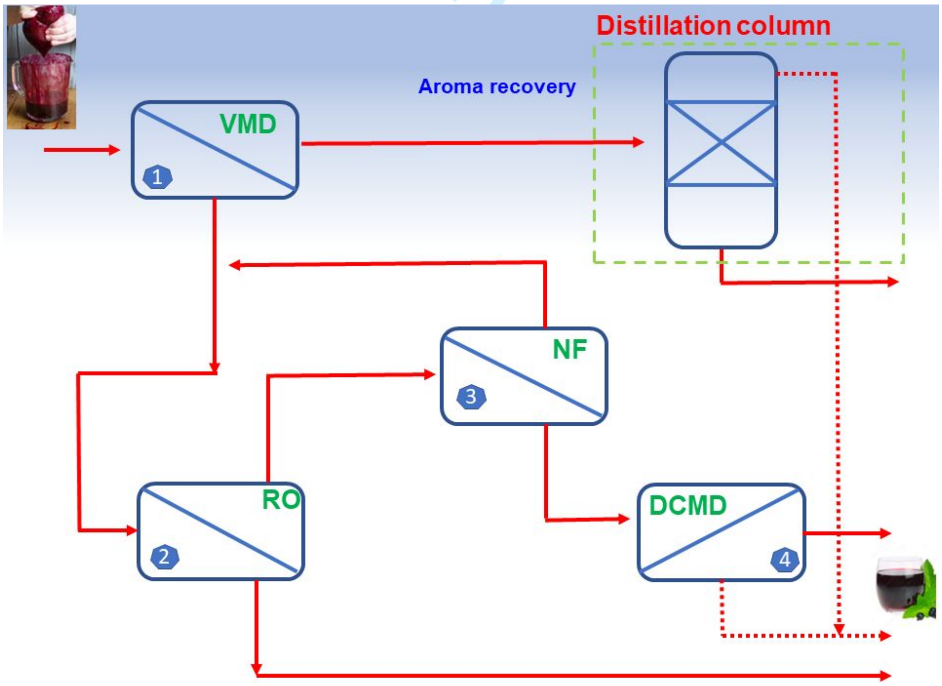


Figure 4. Plant scale membrane processes combined with distillation process developed for the concentration of blackcurrant juice (Sotoft et al., 2012).

In this case, the authors proposed VCMD for the recovery of the aromas, while RO, NF and DCMD processes were used for the water removal by reverse osmosis, nanofiltration and direct contact membrane distillation. Aiming the complete recovery of aroma substances, a distillation column was implemented in such an application. In the plant scale process, the developments had the capacity of processing 20 t/h of raw juice with a production as high as 17,283 ton of concentrated juice yearly. In a further analysis considering the mass balances and process variables (e.g., membrane areas and module numbers), the authors performed a techno economical assessment of the application. The production cost of 0.40 €/kg was determined for the production of concentrated juice (with final 66◦Brix) presenting an initial TSS of 12 ◦Brix. Taking into a count an average membrane lifetime of one year, this latter cost represents 43% lower than that required by the typical thermal evaporation. Pursuing a more economic process, it was considered to extend the membrane life time for 2-3 years (Sotoft et al., 2012). However, to some extent, researchers should be focused on optimizing the operating conditions, e.g., it has reported very recently that VMD operating at vacuum pressure of 10 mbar, the energy consumption can be decreased with an estimated flux decrease from 7 to 5.74 kg/m^{2}h, this point was assessed in juice concentration (Criscuoli, 2022).

Supporting Sotoft's conclusion regarding the economic aspect of MD units compared to conventional thermal evaporation, the quality of the concentrated extract should also be considered. Herein, MD process seems to be also a better option than thermal evaporation, since more nutritional substances are preserved using MD during the concentration. This statements has been documented by Savaş Bahçeci et al. (2015), who observed minimal degradation of vitamin C, preservation of color in the processing of tomato juice. More importantly, the authors also noticed a strong reduction of hydroxymethylfurfural and furan formation, which are undesired chemical substances produced during the thermal treatment of food items. Finally, it seems that fruit juices are the main food items processed by MD technologies; however, some other natural systems, such as ginger (Zou, Hu, et al., 2022; Zou, Kim, et al., 2022) and coffee extracts (Criscuoli & Drioli, 2019), have been subjected to this technology aiming the dehydration and recovery of valuable compounds (such as caffeine). Additionally to this, Tundis et al. (2021) also tested DCMD for the concentration of phenolic substances present in olive mill wastewaters, which is the main by-product of olive oil production. Strategically, the raw extract was preliminary clarified using microfiltration process. After the MD stage, the retentate was reported to contain around five times higher content of hydroxytyrosol and verbascoside compared to the initial extract; and seven times higher content of oleuropein.

Anari et al. (2019) utilized muscadine grape pomace aqueous extracts to recover and concentrate anthocyanins via OD and DCMD. Here, considering the thermolabile properties of anthocyanins, the feed temperature was intentionally restricted to 40 °C while the permeate side was maintained at 10 °C. Particularly, DCMD exhibited a concentration factor of 1.6 but when combining osmotic distillation, the overall concentration factor reached 2.78. In this approach, the outcomes encouraged that MD process should be further investigated for the anthocyanin concentration application. Unfortunately, more interesting outcomes could be obtained with the right selection of the membrane and the possibility of regeneration for further reuse.

3.2. MD processes for the processing of dairy products

MD can also be implemented for other type of applications, such as the treatment of the by-products and effluents from food production processes. For instance, Kezia et al. (2015) treated and concentrated the derived effluent from cheese making industry using DCMD. In this study, a flat sheet PTFE membrane supported on PP non-woven support was implemented. Such a salty whey effluent contained minerals, proteins and sugars requiring a microfiltration stage before DCMD processing. A decrement of feed flux was seen thanks to the presence of trace protein. Herein, the authors concluded that microfiltration was not efficient for the pre-clarification, therefore, a pre-filtration stage implementing UF operation resulted in a more stable flux over 10 h of operation. The process helped to reach a solids concentration up to 30 wt.% initiating from a feed containing 10 wt.% of solids in the feed, while a water recovery of 83 % was achieved.

Proposing a different processes configuration like AGMD, Kujawa et al. (2019) also concentrated lactose and whey solutions from dairy products. In this process, the researchers evaluate the performance of porous hydrophobic membranes (i.e., PP and PTFE), which turned to be effective enough for such concentration (retention index over 99%) and concurrently produce high quality water.

Milk is likely to be the primary dairy product in the food industry. This product has been recently processed by Moejes et al. (2020), who concentrated milk using RO and AGMD. During the optimization protocol, AGMD revealed to be more energy intensive than RO when concentrating the product. According to the author's observations, high energy demand was required to keep the cross flow that need to be heated and cooled. Interestingly, such energy expenditure was dependent on the fouling phenomena. A relevant point declared by the authors towards the performance enhancement of milk concentration relies on the temperature increase of both feed and permeate side until their maximum acceptable value, as well as the reuse of any waste heat.

As documented by Moejes et al. (2020), the fouling becomes a critical aspect due to its direct effect on energy expenditure in MD processes. Here, distinct studies describe and studied such fouling phenomenon taking place during the processing of dairy components by MD (Hausmann, Sanciolo, Vasiljevic, Kulozik, et al., 2013; Hausmann, Sanciolo, Vasiljevic, Weeks, et al., 2013b; Kujawa et al., 2019; Tomaszewska & Białończyk, 2013). Clearly, the formation of fouling layers may cause heat and mass transfer resistances resulting in prominent flux decline (Tijing et al., 2015). By using hydrophobic polytetrafluorethylene (PTFE) membranes, Hausmann et al. (2011) also noticed a flux decay due to the membrane fouling. They analyzed the idea of integrating MD for the processing of different products separately, such as conventional milk, skim milk, whey and a lactose powder; however, they confirmed that the hydrophobic membrane nature promotes the interactions with hydrophobic substances, including fats and proteins, which inherently generate membrane wetting. As for the filtration of whey solution, the fouling was directly associated with time, while in skim milk filtration the fouling was more credited to dry-matter concentration. The fouling issue in MD process has been further analyzed by Hausmann et al. (2013a), who reported that the membrane fouling in skim milk and whey streams initiates by salt and protein deposition. Particularly, for skim milk filtration, the fouling appeared within the first minutes with the formation of homogeneous layer, which got thicker as a function of time. Specific behavior was observed during the filtration of whey, e.g., a formation of fouling patches, which increased their size over the membrane surface and remained reversible. It was observed that this particular fouling layer was less dense resulting in less pronounce flux decay with time.

Facing the fouling issues, change the hydrophobic surfaces into a more hydrophilic ones is considered as a possible pathway to mitigate membrane fouling (Khayet et al., 2006). In this sense, Chanachai et al. (2010) applied as chitosan coating, which is highly hydrophilic (Roberto Castro-Muñoz, González-Valdez, et al., 2020), onto hydrophobic hollow fiber PVDF membranes, which were subsequently used for the concentration of limonene using osmotic distillation. In addition to fouling suppression, the membrane coating contributed in enhanced vapor fluxes while maintaining the flavor. This modified membrane also demonstrated an unchanged flux profile and no wetting signals, while in the pristine PVDF membrane had a drastic flux decay after 100 min testing with signals of wetting issues since $CaCl_2$ has been found in retentate side.

3.3. MD aided processes for the removal of ethanol

In ethanol production, sugar fermentation is performed producing various by-products, which to some extent disturb the yeast activity minimizing the ethanol production in the fermentation reaction, e.g., an ethanol concentration between 5-12%. Theoretically, higher ethanol concentration could minimize its recovery using distillation; unfortunately, such yeast inhibition limited to obtain higher ethanol concentration. At this point, MD represents an economical alternative against distillation, and it can be used for continuous ethanol removal from fermentation systems (Fan et al., 2019; Gryta et al., 2000; Tomaszewska & Białończyk, 2011; Q. Zhang et al., 2017). Importantly, with the ethanol removal, some other polar and volatile substances may be removed as well, which can potentially decrease such inhibitory effect on yeast productivity. Formic, acetic, propionic, butyric, valeric and hexanoic, alcohols (2,3-butanediol), aromatic compounds and furfural are among the volatile compounds generated in fermentation broths (Couallier et al., 2006). Therefore, with this in mind, Gryta (2001) implemented the production of ethanol in a tubular bioreactor coupled with MD. Having a yeast concentration of 20 g/dm^3, the fermentation process assisted by MD was able to produce 5.5 g/dm^{3}h of ethanol, while the same process operated at similar operating conditions but no presenting MD unit produced approximately 2.6 g/dm^{3}h of ethanol, where the overall process efficiency was found to be 50% after 10 h of process. In Gryta's research group, it was confirmed that ethanol, together with volatile substances, can be removed from fermentation broths with MD (Gryta & Barancewicz, 2011). In this case, they noticed that acetic acid and propionic acid were also carried and evaporated from the feed to distillate.

In a recent study, Kumar et al. (2017) experimented bioethanol production integrating MF, NF and DCMD unit operations, as observed in **Figure 5**. This planned strategy was able to operate for several hours with no signal of concentration polarization effect and flux decay. Here, continuous ethanol production from sugarcane juice has been successfully done with high productivity (ca. 9.2 g L^{-1} h^{-1}) and a yield 0.47 g g^{-1}. In this work, the DCMD unit acted as final stage, improving the product purity and concentration enrichment. As an author's suggestion, the integrated membrane system can be operated using solar energy, which can make the process more energy efficient and eco-friendly for ethanol purification.

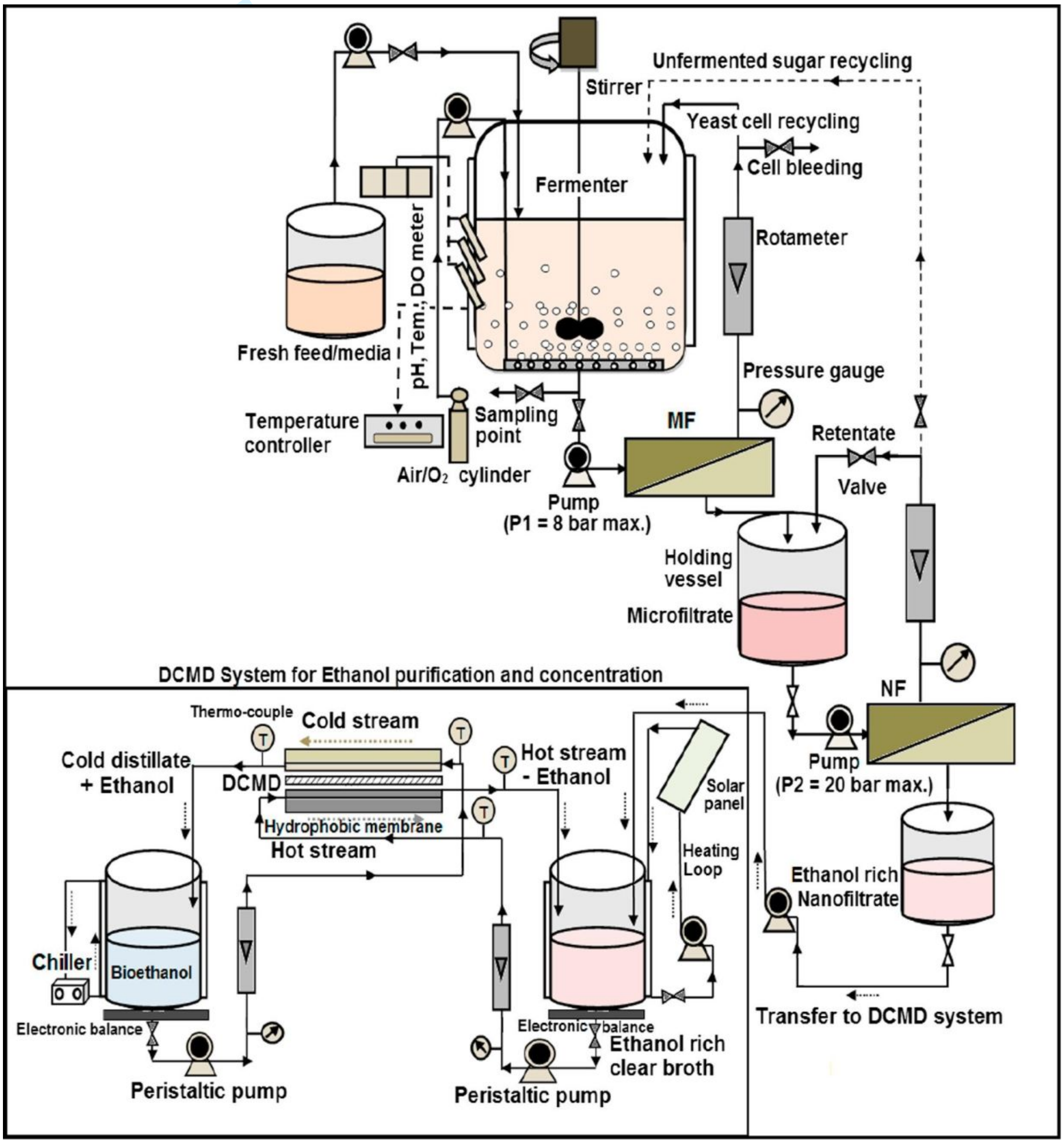


Figure 5. Integration of micro, nanofiltration and membrane distillation for the continuous production of bioethanol. Adapted from (Kumar et al., 2017).

Banat & Al-Shannag (2000), for instance, studied MD for the recovery of diluted acetone-butanol-ethanol in aqueous solution. According to the outcomes reported, the effectiveness of MD process in compensate the inhibitory effect displayed by the acetone-butanol-ethanol on the microbial culture. Also, the feed temperature increase contributed to increase the butanol selectivity, which is indeed highly toxic solvent. Optimizing the process conditions, the optimal feed temperature for butanol extraction was found to be 55 ^{o}C, independently from its high boiling point in comparison with acetone and ethanol.

In the beer manufacturing industries, there is challenge of manufacturing non-alcoholic, or low alcoholic content, beverages from alcoholic beverages (Roberto Castro-Muñoz, 2019). For this task, it is pursued to remove the ethanol once the fermentation in the beer is achieved. Hence, Purwasasmita et al. (2015) attempted beer dealcoholization by proposing VMD. The authors basically studied the effect of pressure of both feed and permeate streams on the flux and selectivity. For the process, a non-porous thin-film composite polyamide (commercial known as TW30-1812-75 from DOW Filmtec from Dow Chemical Company) was equipped. Interestingly, VMD was able to reduce the alcohol content, from 5 to 2.45%-vol., operating within first 6 h. According to author's report, there was no loss of any nutrient and flavoring substances; however, a minimal loss of maltose was observed attributed to the possible adsorption on the membrane surface.

4. Conclusions and perspectives in the field

MD has been successfully applied in processing of foods and food bioactive substances thanks to its lower energy requirement and easy operation compared with traditional distillation. This membrane processes substantially contributes to the preservation of thermal sensitive bioactives molecules, resulting in high quality of products. The main application in food industry deal with the concentration of fruit juices and natural extracts, ethanol removal from alcoholic beverages, processing of dairy products, as well as the recovery and concentration of bioactive substances from food products and wastes. However, this review identifies that most of the applications have been done at lab scale, but promisingly, MD has initiated to be applied in pilot and large scale developments (Sotoft et al., 2012).

As for juice concentration, MD will continue being explored in near future thanks to the high productivities in terms of permeate flu, ranged from 0.6 to 28 L $m^{-2}h^{-1}$ (see **Table 1**). In this latter application, it is likely that PVDF is the most used polymer material for the fabrication of MD with hydrophobic properties. Current scopes of research are still emphasized on improving the properties of this polymer, as well as the resulting properties when using new strategies and items (green solvents) for membrane fabrication (Zou, Hu, et al., 2022; Zou, Kim, et al., 2022). In addition to this last point, a new trend deals with the evaluation of new hydrophobic materials focused on emerging fabrication protocols and hybrid materials, e.g., such as flat sheet electrospun based on PSF/zeolite (Estedlali et al., 2021).

Specific configuration of MD set-ups, such as VMD, are capable to recover aroma substances at the early stages of juice processing (Sotoft et al., 2012). Here, the research on membrane material and properties will be crucial for the control of wetting and fouling in VMD long-term applications.

Over the reviewed applications, the flux decline over time as a consequence of membrane fouling is still one of the main challenges for food processing via MD. To control the membrane fouling, several suggestions can be given, as follows: control the physicochemical properties of the feed, improve the hydrophilic properties of the membranes, or integrate the pre-clarification process prior to MD being applied. Of course, the prominent fouling and wetting of membrane pores negatively influence the membrane performance and durability, limiting the application of MD in this field. For this, research on long-term MD performance is required.

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