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

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3 1 **Membrane distillation assisting the food production processes of thermally**  
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29  
30 12 **Abstract**

31  
32 13 Physical separation technologies have become important tool for processing in the current food  
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34 14 manufacturing industries, especially for the products containing bioactive compounds thanks  
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36 15 to their health benefits in costumers. As for the processing of bioactive food ingredients implies  
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38 16 the implementation of integrated systems oriented to their separation, fractionation, and  
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40 17 recovery. In this field, membrane distillation (MD), which is a thermally driven membrane  
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42 18 process, has been proposed as an alternative for the separation and concentration of liquid food  
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44 19 items. In principle, MD can separate water and volatile compounds from aqueous feed solutions  
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46 20 through a permeate that passes across microporous hydrophobic membranes. The separation  
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48 21 via MD is thanks to the vapor pressure difference on both membrane sides. In this review, we  
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50 22 analyzed the ongoing experimental efforts aimed to recover and purify food bioactive  
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52 23 compounds from the concentration of fruit juices and extracts using MD. Also, the processing  
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54 24 of dairy products, concentration of food by-products, and ethanol production and its removal  
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56 25 from beverages using MD have been reviewed. Additionally, a feedback on the distinct

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3 26 membrane module configurations and membrane requirements for successful operation is  
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5 27 addressed.  
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10 29 **Keywords:** Membrane distillation (MD); thermal driven process; bioactive compounds;  
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12 30 foods; concentration.  
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### 19 33 **1. Introduction**

20  
21 34 Today, separation processes are a core part of agro-food industries since most of the bioactive  
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23 35 substances in nature are tough to obtain in pure form. To some extent, any separation method  
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25 36 is needed before either substance consumption or processing, this is more relevant for functional  
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27 37 molecules and nutraceuticals. Importantly, the proposed method for the processing of food  
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29 38 bioactive components must provide outstanding yield while minimizing the loss and  
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31 39 bioactivity. Within the category of bioactive substances, we can find mostly phenolic  
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33 40 compounds (including anthocyanins, phenolic acids, catechins, flavonones, flavonols, non-  
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35 41 flavonoid polyphenol) carotenoids, oleoresins (capsaicins), polysaccharides, peptides,  
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37 42 terpenoids, phytosterols, among many others (see **Figure 1**). The main concern, which scientists  
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39 43 need to face during the processing of bioactive compounds, relies on their separation from  
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41 44 complex matrix source, and eventually their polishing (purification). As a general overview,  
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43 45 **Figure 1** illustrates a guide of the most typical unit operation required for the pre-treatment,  
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45 46 extraction, purification and concentration stages of bioactive substances contained in natural  
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47 47 sources. Unquestionably, the sequence of separation methods must be raised according to the  
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49 48 type of target analyte and its origin (R. Castro-Muñoz et al., 2022). Additionally, the selected  
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51 49 separation strategy should profit on specific physicochemical property of the substance, such  
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53 50 as size, structure, polarity, etc., which should differ from other molecules present in the source.  
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3 51 In theory, each selected separation or purification technique should effectively perform the  
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5 52 extraction at a low cost, optimal yield minimal loss of compounds (Roberto Castro-Muñoz et  
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7 53 al., 2022; Valencia-Arredondo et al., 2020). Once extracted, the concentration of bioactive-  
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9 54 containing aqueous solutions represents one of the most seek unit operations. In this field,  
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11 55 particular food products require concentration technologies, such as beverages, fruit juices,  
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13 56 vegetable and herbal extracts, milk, whey, etc. Herein, the volume reduction of concentrates  
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15 57 through dewatering critically reduces the substances and water transport, storage and packaging  
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17 58 cost and consequently makes them more resistant to chemical and microbial degradation.  
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61 **Figure 1.** Main bioactive compounds extracted from natural sources and typical extraction  
62 strategy required for their concentration.  
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3 64 To date, typical methods employed for liquid concentration in the food industry, such as  
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5 65 multistage vacuum evaporation, require high energy expenditure, while drastically affecting the  
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7 66 organoleptic and nutritional attributes of the final product due to the high temperature  
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9 67 ([Charcosset, 2021](#); [Echavarría et al., 2011](#)). Over the last decades, many industries have  
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11 68 conceived the raid of membrane technology in particular processes aimed at the separation of  
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13 69 food ingredients. Membrane-based processes introduce multiple advantages during the  
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15 70 processing of natural sources and extraction of bioactive ingredients, such as alternative for  
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17 71 high-energy consuming evaporation process, wastewater and by-products reuse, acceptable  
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19 72 management of wastes, and relatively low capital investments ([Zhong et al., 2021](#)). On the other  
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21 73 hand, issues with membrane durability and maintenance, operating life, replacement costs,  
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23 74 chemical stability, pH sensitivities and fouling issues are among the main drawbacks of  
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25 75 membrane technologies ([Julian et al., 2021](#)). In the field of food processing, membrane  
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27 76 processes, such as microfiltration, ultrafiltration, nanofiltration, forward osmosis, reverse  
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29 77 osmosis and pervaporation have been proposed ([Alvarez et al., 1997](#); [Roberto Castro-Muñoz,](#)  
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31 78 [Boczkaj, et al., 2020](#); [Sant'Anna et al., 2012](#)). Additionally, membrane distillation (MD), which  
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33 79 is defined as thermally driven membrane process, has been ultimately applied for concentration  
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35 80 of foods and natural extracts with interest due to their nutritional value. For instance, MD  
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37 81 operates with lower temperatures and pressures compared with classic distillation process and  
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39 82 even pressure driven membrane unit operations. Furthermore, when treating complex solutions,  
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41 83 the implemented membranes are less prone to fouling comparing to micro and ultrafiltration,  
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43 84 and reverse osmosis ([Lawson & Lloyd, 1996](#); [Onsekizoglu, 2012](#)).

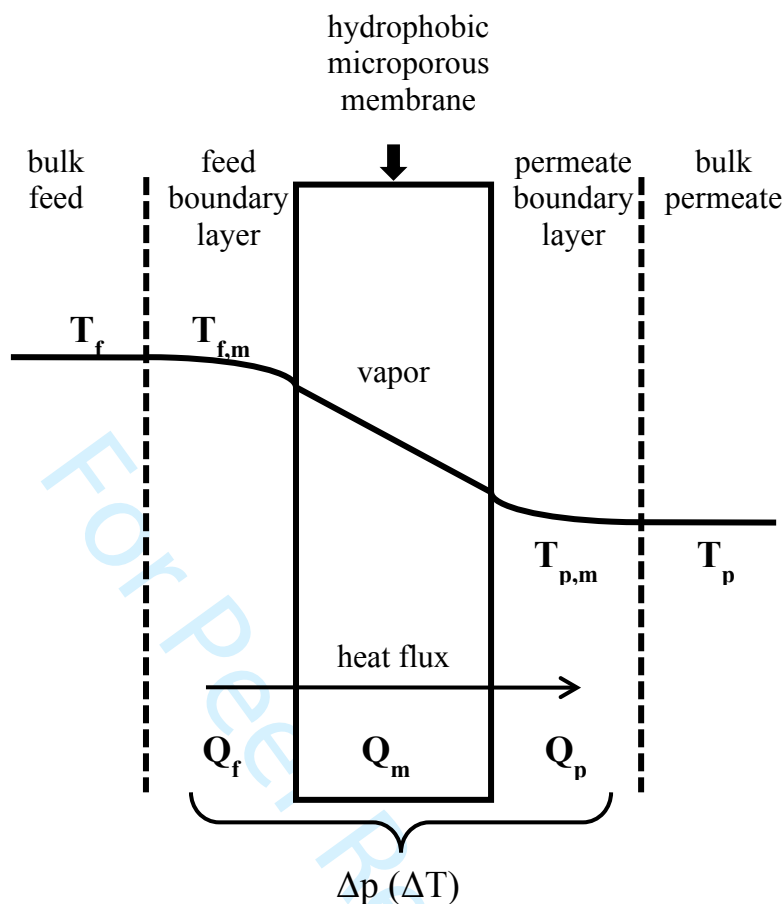
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45 85 With the aim of giving a compelling outlook in this field, this review focuses on covering the  
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47 86 most relevant application of MD in the food industry in terms of clarification and concentration  
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49 87 of juices and natural extracts. Concurrently, feedback on theoretical principles and parameters  
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51 88 influencing this process is also given, along with the main process parameters involved in the  
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3 89 production of such food items. Apart from this, we present some advantages and disadvantages  
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5 90 of MD technology compared with other membrane processes.  
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10 92 **2. Thermal-driven membrane distillation: Principles, process variables and**  
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12 93 **membrane requirements**

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14 94 Membrane distillation (MD) uses a vapour pressure gradient as the driving force, which is  
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17 95 created between feed and permeate streams by inducing a temperature difference across the  
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19 96 permeable and selective physical barrier (i.e. membrane). Both streams are in direct contact  
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21 97 with the membrane that may presents a microporous structure. Typically, the membrane must  
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23 98 own a hydrophobic character, which will fundamentally prevent the transport of the feed bulk  
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25 99 in liquid state. At each membrane pore, liquid/gas interface is generated, in which the vapour  
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27 100 phase permeates through the membrane and later condensate at the permeate stream.

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30 101 The mass and heat transfer concurrently takes place. In theory, two relevant heat transfer  
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32 102 mechanisms use to appear. For instance, the conductive heat transfer inside the membrane pores  
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34 103 takes place along with the vapor diffusion, resulting in a temperature change at both membrane  
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36 104 boundary layers. This latter phenomenon produces a temperature gradient between the feed and  
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38 105 permeate sides and hence origins a convective heat transfer. **Figure 2** illustrates the heat flux  
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40 106 in direct contact MD process.  
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**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.,

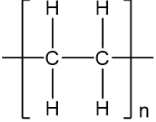
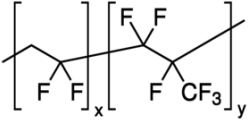
2016; H. Zhang et al., 2018). Here, we describe some issues and desired properties in membranes for MD technology.

- 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 <sup>-3</sup> Nm <sup>-1</sup> )	LEP	Reference:
PVDF	$\left[ \begin{array}{cc} \text{H} & \text{F} \\   &   \\ -\text{C} & -\text{C}- \\   &   \\ \text{H} & \text{F} \end{array} \right]_n$	113°	30.3	21.3 kPa	(J. Zhang et al., 2010)
PTFE	$\begin{array}{cc} \text{F} & \text{F} \\   &   \\ \text{R}-\text{C} & -\text{C}-\text{R} \\   &   \\ \text{F} & \text{F} \end{array}$	126°	9.1	24 kPa	(J. Zhang et al., 2010)
PP	$\left[ \begin{array}{c} \text{CH}_3 \\   \\ -\text{CH}-\text{CH}_2- \\   \\ \text{H} \end{array} \right]_n$	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)

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- 140 • *Membrane thickness*: This property affects the transport overall MD process in an

141 inversely proportional way. For example, thinner membranes reduce the mass transfer

142 resistance and thus increase the vapor flux. On the contrary, the membrane thickness

143 affects the conductive heat loss over the MD operation. In this way, the implemented

144 membrane should preferentially be as thick as possible to mitigate the heat loss.

145 Unfortunately, this latter point compromises the high vapour flux requirement;

146 therefore, the thickness needs to be optimized, e.g., it has been documented that the

147 optimal membrane thickness for a MD process should be ranged from 30 to 60  $\mu\text{m}$

148 (Laganà et al., 2000).
- 149 • *Membrane porosity*: This parameter is determined as a relationship between volume of

150 the pores and the total membrane volume. Importantly, the membrane porosity is

151 directly proportional to the evaporation surface area, thus, higher porosity of the

152 membrane results into higher vapor flux (Susanto, 2011). In addition to this, a

153 membrane porosity increase substantially reduces the conductive heat loss, as the

154 conductive heat transfer coefficient of the gases entrapped in the membrane pores is

155 typically an order of magnitude smaller than the conductive heat transfer coefficient of

156 the hydrophobic membrane material (Lawson & Lloyd, 1996). For instance, it has been

157 determined that the membrane porosity value for an exceptional MD must be ranged

158 from 30 to 85% (El-Bourawi et al., 2006).

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3 159 • *Membrane pore size*: As stated early, microporous structure in membranes is typically  
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5 160 needed for MD. Also, the membrane pore size requires be optimized to prevent wetting  
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7 161 of the membrane, and concurrently, ensure the highest flux. To some extent, the optimal  
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9 162 pore size depends on the purpose of the application, which intrinsically depends on the  
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11 163 kind of feed solution. Theoretically, [Schneider et al. \(1988\)](#) declared a pore diameter  
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13 164 ranged from 0.5 to 0.6  $\mu\text{m}$  seems to be enough to mitigate the wetting. Of course,  
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15 165 uniform pore size over the membrane structure is desired to get a better approximation  
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17 166 in terms of vapour flux ([Susanto, 2011](#)).
- 18  
19 167 • *Pore tortuosity*: Together with the pore size, the shape of membrane pores also plays a  
20  
21 168 great role in the membrane process. As a definition, the membrane tortuosity represents  
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23 169 the pore shape deviation from the cylindrical structure. This property is given by the  
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25 170 average length of the pores in comparison with the membrane thickness. In terms of  
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27 171 separation performance, when the membrane pores make tortuous pathways, there is a  
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29 172 decrement in the flux of the diffusing molecules, therefore, low tortuosity results in high  
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31 173 vapor flux. Commonly, the most frequent tortuosity is 2 ([Phattaranawik et al., 2003](#)).
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33 174 • *Thermal conductivity*: Since we seek minimizing the heat loss in MD, it is obvious to  
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35 175 prepare membranes based on a material with low heat conductivity. The heat loss  
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37 176 decrement results in higher energy efficiency, lower susceptibility to temperature  
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39 177 polarization phenomena, as well as higher vapor flux. As mentioned previously, the  
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41 178 membrane thermal conductivity is generally higher than that of gases occupying the  
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43 179 pores. It is known that the thermal conductivities of distinct polymers, such as PP, PTFE,  
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45 180 PVDF, are ranged from 0.11 up to 0.27  $\text{Wm}^{-1}\text{K}^{-1}$  at 23  $^{\circ}\text{C}$  ([Alkhudhiri et al., 2012](#)).

53  
54 181 Summarizing such membrane properties, **Table 2** gives the characterization data of typical  
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56 182 commercial membranes applied in MD. It is worth mentioning that most of these commercial  
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58 183 membranes do not meet all the MD requirements or lacking in any of such properties. Therefore,  
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184 there is a necessity of designing outperforming membranes with desirable membrane features.

185 Apart from these latter aspects, there is also a specific drawback in hydrophobicity nature

186 improvement of smooth membrane surfaces. This is the case of PVDF, which displays a smooth

187 surface as a result of saturation by fluorinated methyl groups, in general, the membranes are

188 able to achieve a maximum water contact angle of 120° (Liao et al., 2013). In this sense, a

189 feasible pathway for improving the surface hydrophobicity is through the increase of surface

190 roughness (Tijing et al., 2016). Apart from reaching a strong water repellence, a particular

191 attention should be focused to design a micro- and nanostructured surface as well. Here,

192 ongoing research has utilized nanofillers to be embedded into the polymer membrane to tailor

193 the physicochemical properties and structure of membranes, including hydrophobicity,

194 porosity, chemical, thermal and mechanical stability, and surface charge density. This

195 membrane modification by filler loading is named the new generation of membranes, in which

196 different nanomaterials have been used including carbon nanotubes (Tijing et al., 2016),

197 graphene (Gontarek et al., 2019), clay (Prince et al., 2012), silica (Y. Zhang & Wang, 2013)

198 and titanium dioxide (Meng et al., 2014). Depending on the nanoparticle's properties,

199 superhydrophobic MD membranes can also be obtained.

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**Table 2.** Properties of commercial MD membranes.

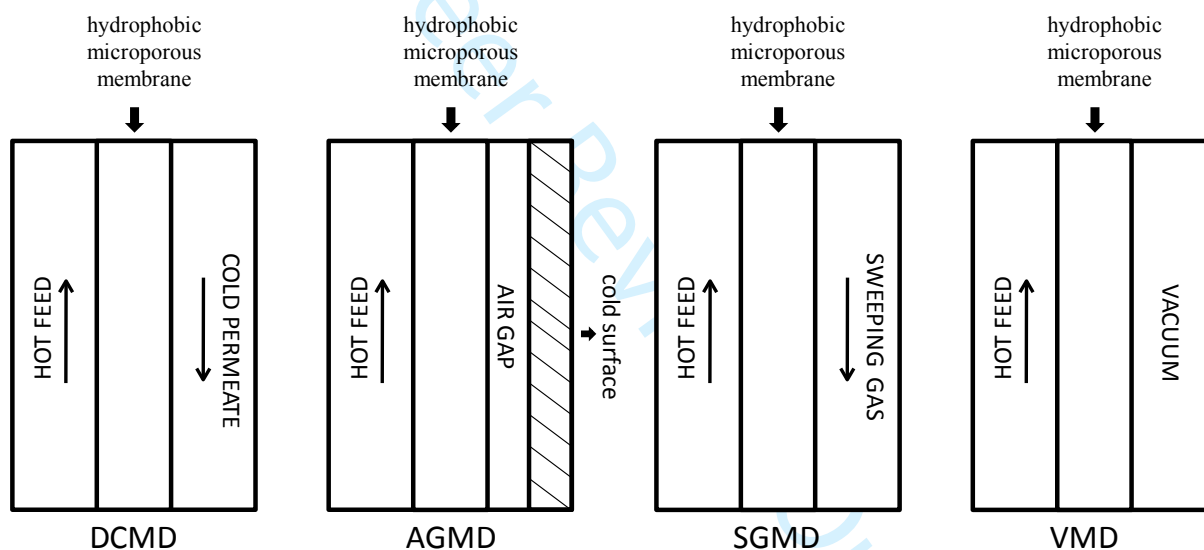
Polymer/module:	Trade name:	Manufacturer:	Thickness ( $\mu\text{m}$ ):	Mean pore size ( $\mu\text{m}$ ):	Porosity (%):
PVDF/flat sheet	GVHP	Milipore	125	0.2	80
PVDF/flat sheet	Durapore	Milipore	110	0.45	75
PTFE/flat sheet		Osmonics	175	0.22	70
PTFE/hollow fiber	POREFLON	Sumitomo Electric	550	0.8	62

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	-

202

## 203 2.2. MD operation configurations

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



208

209 **Figure 3.** Different operation configuration of MD.

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- 211 • Direct contact membrane distillation (DCMD): In this configuration both feed and  
 212 permeate liquids are in direct contact with the hydrophobic microporous membrane.  
 213 Since the permeate presents lower temperature than that of the feed, permeate is  
 214 intentionally employed for the condensation of the other fluid. Thanks to its easy  
 215 implementation and simplicity, this MD configuration is the most common  
 216 implemented at lab scale. Unfortunately, the obvious direct contact of liquids with

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3 217 membrane interface results into a heat loss over the membrane. At this point, DCMD is  
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5 218 identified as the lowest thermal efficiency among all MD operation configurations.  
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8 219 • Air gap membrane distillation (AGMD): In this mode, the vapour phase permeating  
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10 220 across the membrane is condensed onto a cold surface. Herein, the membrane and cold  
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12 221 surface are intentionally separated from each other by an air gap. In contrast to DCMD,  
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14 222 AGMD displays less heat loss over the membrane.  
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17 223 • Sweeping gas membrane distillation (SGMD): Here, the vapour permeating is sweeping  
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19 224 and carrying by using a cold inert gas outside the membrane, which is finally condensed.  
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21 225 The low heat loss and reduced mass transfer are among the main advantages of this MD  
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23 226 configuration, while the high operational cost compared with other configurations is  
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25 227 identified as its main drawback making to be used rarely.  
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28 228 • Vacuum membrane distillation (VMD): In this configuration, the permeate stream is  
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30 229 subjected to a vacuum that induces a driving force. Thanks to the low pressure,  
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32 230 condensation phenomenon occurs outside of the membrane.  
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### 37 38 232 2.3. Types of membrane modules and requirements

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40 233 To date, several membrane modules have been successfully designed for MD, such as plate and  
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42 234 frame, spiral-wound, tubular, capillary or hollow fiber (Alkhudhiri et al., 2012). A typical  
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44 235 membrane module aimed to be used in MD should display a high packing density. This latter  
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46 236 property denotes the ratio of membrane area to the packing volume. Additionally, the  
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48 237 membrane must offer high feed and permeate flow rates, which are commonly fed tangentially  
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50 238 to the membrane (or also denominated as *crossflow* mode). Interestingly, the module design  
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52 239 should potentially offer the chance of creating turbulent flow rate to give heat and mass transfer  
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54 240 among bulk solution and the solution at boundary layer. In this sense, prevention of the  
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56 241 temperature polarization and concentration polarization effects can be observed.  
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3 242 As part of required characteristic for MD membranes, the module needs to provide a low  
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5 243 pressure drop over the module length to mitigate the flooding of membrane pores, which is  
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7 244 generally caused by a high transmembrane hydrostatic pressure. Also, the membrane module  
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9 245 should guarantee unchanged temperature profile of the liquids over the module length, together  
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11 246 with minimal heat loss. Regarding the membrane material, it should be selected considering the  
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13 247 treated feed solutions that can compromise the structural properties of the membranes, e.g.,  
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15 248 corrosion.  
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### 21 250 **3. Findings on MD processes for the processing of food items**

#### 22 251 *3.1. Clarification and concentration of natural extracts and fruit juices*

23  
24 252 As most of the membrane unit operations, MD can be operated with mild temperatures and  
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26 253 atmospheric pressures; these operational conditions make it suitable for juice concentration  
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28 254 containing thermolabile bioactive substances (Bhattacharjee et al., 2017). In other words, this  
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30 255 process can potentially minimize the degradation of such high-added value molecules. In  
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32 256 pioneering works, such hypothesis has been proven by Calabrò et al. (1994), who evaluated  
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34 257 the performance of microporous PVDF membrane for the concentration of orange juice. The  
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36 258 researchers noted exceptional retention of different molecules, such as soluble solids, sugars,  
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38 259 and organic acids. Considering such findings, the research community supported the  
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40 260 exploration of this membrane technology confirming it as a potential alternative aiming the  
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42 261 concentration of distinct fruit juices, including orange (Deshmukh et al., 2011), apple (Gunko  
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44 262 et al., 2006), black currant (Bagger-Jørgensen et al., 2004), kiwi fruit (A. Cassano & Drioli,  
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46 263 2007), pineapple (Hongvaleerat et al., 2008), grape juices (Rektor et al., 2006), to mention just  
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48 264 a few. **Table 3** enlists the main applications of MD dealing with the concentration of fruit  
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50 265 juices, applied operating conditions and unit operation configuration and the most relevant  
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52 266 outcomes.  
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**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/m <sup>2</sup> /s *10 <sup>3</sup>	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 <sup>2</sup> h	65 °Brix	(Quist-Jensen et al., 2016)
DCMD	Tf : 70 °C Tp : 10 °C	flat sheet PVDF	apple juice	28 Lm <sup>-2</sup> h <sup>-1</sup>	to 60–65 °Brix	(Gunko et al., 2006)
OD	Tf : 35 °C Tp : 20 °C	flat sheet PTFE	pineapple	10 kg/m <sup>2</sup> h	10.6 to 27.8 g 100 g <sup>-1</sup> TSS	(Hongvaleerat et al., 2008)
DCMD	Tf : 30 °C Tp : 11 °C	hollow fiber PP	black currant	0.8 kg/m <sup>2</sup> h	58.2 °Brix	(Kozák et al., 2009)
DCMD	Tf : 32 °C Tp : ~4 °C	hollow fiber PP	apple juice	1 kg/m <sup>2</sup> h	64°Brix	(Laganà et al., 2000)
OMD	Tf : 35 °C Tp : 20 °C	flat sheet PTFE	cactus pear juice	3-4 Lm <sup>-2</sup> h <sup>-1</sup>	23.4 °Brix	(Terki et al., 2018)
DCMD	Tf : 30 °C Tp : -	capillary PP	grape juice	2-2.5 kg/m <sup>2</sup> h	65 °Brix	(Rektor et al., 2006)
OD	Tf : 40 °C Tp : 40 °C	hollow fiber PP	cranberry juice	1.21 Lm <sup>-2</sup> h <sup>-1</sup>	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 <sup>-2</sup> h <sup>-1</sup>	40 °Brix	Savaş Bahçeci et al. (2015)

DCMD	Tf : 35 °C Tp : 20 °C	flat sheet PVDF	Propolis extract	0.94 kg m <sup>-2</sup> h <sup>-1</sup>	-	(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)

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270

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

284 Bagger-Jørgensen et al. (2011) evaluated two MD configurations in recovering aroma  
 285 molecules from black currant. Initially, the authors investigated the influence on key operation  
 286 parameters, such as feed temperature and feed flow, on the permeate flux and concentrate  
 287 properties and quality. Here, twelve aroma substances were targeted for the determination of



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3 288 concentration factor, which in fact they were found to be the highest at the highest feed  
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5 289 temperature. For instance, the process showed that for the volatile aromas with the most  
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7 290 hydrophobic nature displayed concentration factors ranged from 9.3 up to 12.1 (at 45°C). Also,  
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9 291 the highest operating temperature of the feed and its flow rate of 400 L/h revealed an aroma  
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11 292 recovery of approximately 84 vol.%. It is important to note that the researchers made a  
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13 293 comparison with the recovery efficiency between SGMD and VMD. In general, SGMD process  
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15 294 seemed to be less affected by the flow rate but was more impacted by the temperature. While  
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17 295 the concentration by VMD configuration shortened the operating time since the higher flux was  
18  
19 296 obtained. On the contrary, as the longer time demanded by SGMD for the concentration, a  
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21 297 higher loss of bioactive substances, such as anthocyanins and polyphenols, was acquired in  
22  
23 298 comparison with the VMD.

24  
25  
26 299 In a different work, [Onsekizoglu et al. \(2010\)](#) also used MD for apple juice concentration,  
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28 300 revealing that the feed flow rate had null effect on the transmembrane flux as compared with  
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30 301 of temperature difference effect. In the MD operation, the initial clarified apple juice presented  
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32 302 total soluble solids (TSS) of 12 °Brix, a value which was reached to be up to 65 °Brix after  
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34 303 concentration. Regarding the impact of the process on the juice quality, both nutritional and  
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36 304 sensorial properties remained like the original juice. The authors concluded that MD, unlike  
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38 305 conventional thermal evaporation, contributed to maintaining concentrated juice's natural color  
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40 306 appearance and attractive aroma.

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43 307 As a key principle, it is crucial to control the constant trans-membrane vapor pressure to  
44  
45 308 mitigate a loss in permeate flux in MD ([Laganà et al., 2000](#)). In the line of this analysis, [Quist-](#)  
46  
47 309 [Jensen et al. \(2016\)](#) in their work concentrated a clarified orange juice via DCMD. The  
48  
49 310 researchers observed an evaporation flux decrement at the preconcentration step, which was  
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51 311 credited by the decrease in temperature difference between the feed and permeate. Surprisingly,  
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53 312 the outcomes declared that in such final concentration stage, the trans-membrane flux  
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3 313 decrement can also be influenced by the juice viscosity increase. Testing two-step DCMD  
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5 314 process, the TSS in clarified orange juice has increased from 9.5 up to 65 °Brix. Supporting  
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7 315 Jensen's results, [Gunko et al. \(2006\)](#) also experimented a significant temperature gradient  
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9 316 dependence on the DCMD operation. The outcomes exhibited that the cooling water  
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11 317 temperature decrease, from 30°C to 10°C, improved the flux ca. two times (180%) when  
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13 318 operating at 50 °C. Unfortunately, the same decrease in cooling water temperature for the feed  
14  
15 319 temperature equal 70°C resulted in only 10% flux improvement. In terms of the best permeate  
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17 320 flux (ca. 28 LMH), this was observed at the beginning of the concentration process. Once the  
18  
19 321 concentration initiated to be reached, there was a decrement in permeate flux, e.g., when TSS  
20  
21 322 content was 50 °Brix, the permeate flux decreased until 9 L/m<sup>2</sup>/h, which continued to decrease  
22  
23 323 until reaching a concentration between 60–65 °Brix. Similar TSS (ca. 64 °Brix) was obtained  
24  
25 324 by [Laganà et al. \(2000\)](#), who proposed polypropylene hollow-fiber modules for DCMD  
26  
27 325 configuration aimed to concentrate apple juices. In this work, the authors noticed that flux was  
28  
29 326 majorly dependent on temperature polarization coefficient, rather than concentration  
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31 327 polarization coefficient which was found to be negligible.  
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40 329 An important constraint in the concentration of fruit juices using membrane technologies (like  
41  
42 330 MD) relies on the membrane fouling. This latter phenomenon occurs due to the content of  
43  
44 331 colloidal particles in the extract which may cause clogging or blocking of membrane pores  
45  
46 332 ([Pichardo-Romero et al., 2020](#)). For instance, [Mirsaeedghazi et al. \(2009\)](#) eventually noted cake  
47  
48 333 layer formation on the membrane surface which inevitably caused serious membrane fouling,  
49  
50 334 such event occurred within the first 5 min of operation during the processing of pomegranate  
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52 335 juice. Apart from, the membrane fouling, the layer deposited also promoted the membrane  
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54 336 wetting and thus resulted in the transport of the liquid across the membrane. Usually,  
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3 337 carbohydrate foulants, such as cellulose, pectins, lignin and hemicelluloses, are found in fruit  
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5 338 juices.

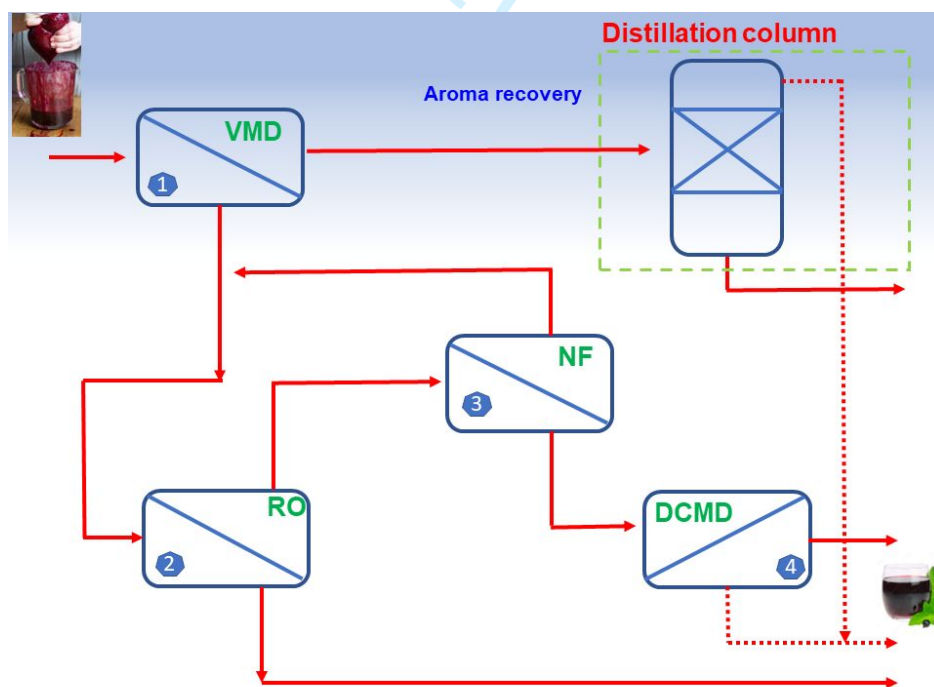
7 339 Knowing the effect of membrane fouling on the process operation of MD modules, the  
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10 340 researchers started to face membrane fouling by strategically integrating different membrane  
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12 341 unit operations and pre-treatments. For example, [He et al. \(2007\)](#) clearly observed that the pre-  
13  
14 342 clarification of the apple juice helped to obtained higher productivity in terms of permeate flux  
15  
16 343 in UF process. Here, enzymatic pretreatment has been proposed as initial step of juice  
17  
18 344 clarification by hydrolyzing the pectin and other polysaccharides ([Onsekizoglu et al., 2010](#)).  
19  
20 345 After such pre-treatments and clarification using membranes, MD can be implemented  
21  
22 346 obtaining more efficient and less prone to fouling processes. The preliminary filtration is  
23  
24 347 typically suggested to remove suspended solids and macromolecules from the fruit juices,  
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26 348 which consequently contributes to reduce the extract viscosity and thus acquire higher fluxes  
27  
28 349 during MD concentration stage. It is known that the reduction of juice viscosity enhances  
29  
30 350 hydrodynamic conditions in the membrane pores and therefore decreases the concentration and  
31  
32 351 temperature polarization phenomena ([Lukanin et al., 2003](#)).

33 352 Integrating macromolecular sieving clarification (like MF) with MD, [Rektor et al. \(2006\)](#)  
34  
35 353 clarified and concentrated grape juice, respectively, in which the final concentrate extract was  
36  
37 354 subjected to a subsequent water implementing another membrane technique, such as reverse  
38  
39 355 osmosis (RO). In a more recent development, [Onsekizoglu \(2013\)](#) combined UF and MD  
40  
41 356 operations to concentrate pomegranate juice evaluating their impact on product quality and  
42  
43 357 process performance. Initially, the UF stage contributed to improve the physical aspect of the  
44  
45 358 juice while maintaining valuable substances such as organic acids. The ultrafiltration confirmed  
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47 359 again the removal of macromolecular compounds, which may consequently provoke membrane  
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49 360 wetting and can also produce a non-allowable convective flow of liquid through the post  
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51 361 treatment with the MD membrane. Overall, integrated membrane operations permitted to  
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362 produce a concentrated pomegranate juice containing 57 °Brix with acceptable content of  
 363 phenolic compounds, total monomeric anthocyanins, pH and color.

364 Other membrane technologies, such as reverse and forward osmosis, have been utilized for the  
 365 pre-concentration stages of natural extract prior MD. Several cases of study haven documented  
 366 proposing such processing strategy, e.g., acerola juice (Pagani et al., 2011), blood orange  
 367 (Galaverna et al., 2008), and citrus and carrot juices (A. Cassano et al., 2003). Implementing  
 368 three different membrane stages, Kozák et al. (2009) successfully concentrated blackcurrant  
 369 juice concentration using DCMD, however, the initial extract was firstly clarified with MF,  
 370 followed by pre-concentration via reverse osmosis. Both membrane processes (MF and RO)  
 371 contributed to concentrate the juice from 15 to 22 °Brix, afterwards, DCMD greatly  
 372 concentrated the extract until 58 °Brix by applying only a temperature difference of 19 °C.

373 Similar to Kozak's study, Sotoft et al. (2012) also concentrated blackcurrant juice integrating  
 374 various membrane operations, as described in **Figure 4**.



375  
 376 **Figure 4.** Plant scale membrane processes combined with distillation process developed for the  
 377 concentration of blackcurrant juice (Sotoft et al., 2012).

378

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

395 Supporting Sotoft's conclusion regarding the economic aspect of MD units compared to  
396 conventional thermal evaporation, the quality of the concentrated extract should also be  
397 considered. Herein, MD process seems to be also a better option than thermal evaporation, since  
398 more nutritional substances are preserved using MD during the concentration. This statements  
399 has been documented by Savaş Bahçeci et al. (2015), who observed minimal degradation of  
400 vitamin C, preservation of color in the processing of tomato juice. More importantly, the authors  
401 also noticed a strong reduction of hydroxymethylfurfural and furan formation, which are  
402 undesired chemical substances produced during the thermal treatment of food items. Finally, it

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3 403 seems that fruit juices are the main food items processed by MD technologies; however, some  
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5 404 other natural systems, such as ginger (Zou, Hu, et al., 2022; Zou, Kim, et al., 2022) and coffee  
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7 405 extracts (Criscuoli & Drioli, 2019), have been subjected to this technology aiming the  
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9 406 dehydration and recovery of valuable compounds (such as caffeine). Additionally to this,  
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11 407 Tundis et al. (2021) also tested DCMD for the concentration of phenolic substances present in  
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13 408 olive mill wastewaters, which is the main by-product of olive oil production. Strategically, the  
14  
15 409 raw extract was preliminary clarified using microfiltration process. After the MD stage, the  
16  
17 410 retentate was reported to contain around five times higher content of hydroxytyrosol and  
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19 411 verbascoside compared to the initial extract; and seven times higher content of oleuropein.  
20  
21 412 Anari et al. (2019) utilized muscadine grape pomace aqueous extracts to recover and  
22  
23 413 concentrate anthocyanins via OD and DCMD. Here, considering the thermolabile properties of  
24  
25 414 anthocyanins, the feed temperature was intentionally restricted to 40 °C while the permeate side  
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27 415 was maintained at 10 °C. Particularly, DCMD exhibited a concentration factor of 1.6 but when  
28  
29 416 combining osmotic distillation, the overall concentration factor reached 2.78. In this approach,  
30  
31 417 the outcomes encouraged that MD process should be further investigated for the anthocyanin  
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33 418 concentration application. Unfortunately, more interesting outcomes could be obtained with the  
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35 419 right selection of the membrane and the possibility of regeneration for further reuse.  
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### 44 421 3.2. MD processes for the processing of dairy products

46 422 MD can also be implemented for other type of applications, such as the treatment of the by-  
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48 423 products and effluents from food production processes. For instance, Kezia et al. (2015) treated  
49  
50 424 and concentrated the derived effluent from cheese making industry using DCMD. In this study,  
51  
52 425 a flat sheet PTFE membrane supported on PP non-woven support was implemented. Such a  
53  
54 426 salty whey effluent contained minerals, proteins and sugars requiring a microfiltration stage  
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56 427 before DCMD processing. A decrement of feed flux was seen thanks to the presence of trace  
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3 428 protein. Herein, the authors concluded that microfiltration was not efficient for the pre-  
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5 429 clarification, therefore, a pre-filtration stage implementing UF operation resulted in a more  
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7 430 stable flux over 10 h of operation. The process helped to reach a solids concentration up to 30  
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9 431 wt.% initiating from a feed containing 10 wt.% of solids in the feed, while a water recovery of  
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11 432 83 % was achieved.

12  
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14 433 Proposing a different processes configuration like AGMD, [Kujawa et al. \(2019\)](#) also  
15  
16 434 concentrated lactose and whey solutions from dairy products. In this process, the researchers  
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18 435 evaluate the performance of porous hydrophobic membranes (i.e., PP and PTFE), which turned  
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20 436 to be effective enough for such concentration (retention index over 99%) and concurrently  
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22 437 produce high quality water.

23  
24  
25 438 Milk is likely to be the primary dairy product in the food industry. This product has been  
26  
27 439 recently processed by [Moejes et al. \(2020\)](#), who concentrated milk using RO and AGMD.  
28  
29 440 During the optimization protocol, AGMD revealed to be more energy intensive than RO when  
30  
31 441 concentrating the product. According to the author's observations, high energy demand was  
32  
33 442 required to keep the cross flow that need to be heated and cooled. Interestingly, such energy  
34  
35 443 expenditure was dependent on the fouling phenomena. A relevant point declared by the authors  
36  
37 444 towards the performance enhancement of milk concentration relies on the temperature increase  
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39 445 of both feed and permeate side until their maximum acceptable value, as well as the reuse of  
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41 446 any waste heat.

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43  
44 447 As documented by [Moejes et al. \(2020\)](#), the fouling becomes a critical aspect due to its direct  
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46 448 effect on energy expenditure in MD processes. Here, distinct studies describe and studied such  
47  
48 449 fouling phenomenon taking place during the processing of dairy components by MD  
49  
50 450 ([Hausmann, Sancio, Vasiljevic, Kulozik, et al., 2013](#); [Hausmann, Sancio, Vasiljevic,](#)  
51  
52 451 [Weeks, et al., 2013b](#); [Kujawa et al., 2019](#); [Tomaszewska & Białończyk, 2013](#)). Clearly, the  
53  
54 452 formation of fouling layers may cause heat and mass transfer resistances resulting in prominent  
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3 453 flux decline (Tijing et al., 2015). By using hydrophobic polytetrafluorethylene (PTFE)  
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5 454 membranes, Hausmann et al. (2011) also noticed a flux decay due to the membrane fouling.  
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7  
8 455 They analyzed the idea of integrating MD for the processing of different products separately,  
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10 456 such as conventional milk, skim milk, whey and a lactose powder; however, they confirmed  
11  
12 457 that the hydrophobic membrane nature promotes the interactions with hydrophobic substances,  
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14 458 including fats and proteins, which inherently generate membrane wetting. As for the filtration  
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17 459 of whey solution, the fouling was directly associated with time, while in skim milk filtration  
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19 460 the fouling was more credited to dry-matter concentration. The fouling issue in MD process  
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21 461 has been further analyzed by Hausmann et al. (2013a), who reported that the membrane fouling  
22  
23 462 in skim milk and whey streams initiates by salt and protein deposition. Particularly, for skim  
24  
25 463 milk filtration, the fouling appeared within the first minutes with the formation of homogeneous  
26  
27 464 layer, which got thicker as a function of time. Specific behavior was observed during the  
28  
29 465 filtration of whey, e.g., a formation of fouling patches, which increased their size over the  
30  
31 466 membrane surface and remained reversible. It was observed that this particular fouling layer  
32  
33 467 was less dense resulting in less pronounce flux decay with time.  
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36  
37 468 Facing the fouling issues, change the hydrophobic surfaces into a more hydrophilic ones is  
38  
39 469 considered as a possible pathway to mitigate membrane fouling (Khayet et al., 2006). In this  
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41 470 sense, Chanachai et al. (2010) applied as chitosan coating, which is highly hydrophilic (Roberto  
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43 471 Castro-Muñoz, González-Valdez, et al., 2020), onto hydrophobic hollow fiber PVDF  
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45 472 membranes, which were subsequently used for the concentration of limonene using osmotic  
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47 473 distillation. In addition to fouling suppression, the membrane coating contributed in enhanced  
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49 474 vapor fluxes while maintaining the flavor. This modified membrane also demonstrated an  
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51 475 unchanged flux profile and no wetting signals, while in the pristine PVDF membrane had a  
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53 476 drastic flux decay after 100 min testing with signals of wetting issues since  $\text{CaCl}_2$  has been  
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55 477 found in retentate side.  
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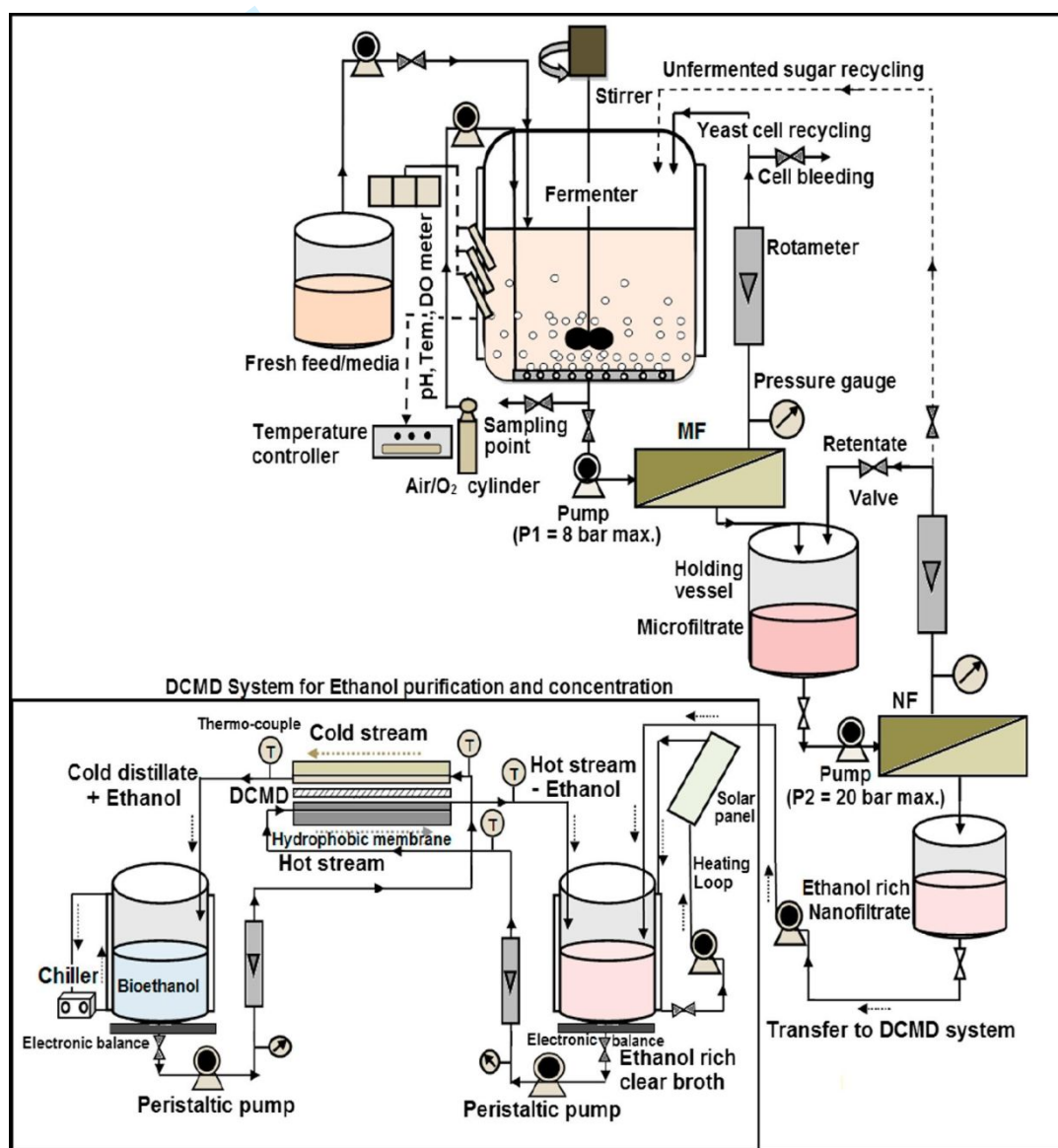


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5 479 *3.3. MD aided processes for the removal of ethanol*

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7 480 In ethanol production, sugar fermentation is performed producing various by-products, which  
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9 481 to some extent disturb the yeast activity minimizing the ethanol production in the fermentation  
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11 482 reaction, e.g., an ethanol concentration between 5-12%. Theoretically, higher ethanol  
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13 483 concentration could minimize its recovery using distillation; unfortunately, such yeast  
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15 484 inhibition limited to obtain higher ethanol concentration. At this point, MD represents an  
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17 485 economical alternative against distillation, and it can be used for continuous ethanol removal  
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19 486 from fermentation systems (Fan et al., 2019; Gryta et al., 2000; Tomaszewska & Białończyk,  
20  
21 487 2011; Q. Zhang et al., 2017). Importantly, with the ethanol removal, some other polar and  
22  
23 488 volatile substances may be removed as well, which can potentially decrease such inhibitory  
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25 489 effect on yeast productivity. Formic, acetic, propionic, butyric, valeric and hexanoic, alcohols  
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27 490 (2,3-butanediol), aromatic compounds and furfural are among the volatile compounds  
28  
29 491 generated in fermentation broths (Couallier et al., 2006). Therefore, with this in mind, Gryta  
30  
31 492 (2001) implemented the production of ethanol in a tubular bioreactor coupled with MD. Having  
32  
33 493 a yeast concentration of 20 g/dm<sup>3</sup>, the fermentation process assisted by MD was able to produce  
34  
35 494 5.5 g/dm<sup>3</sup>h of ethanol, while the same process operated at similar operating conditions but no  
36  
37 495 presenting MD unit produced approximately 2.6 g/dm<sup>3</sup>h of ethanol, where the overall process  
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39 496 efficiency was found to be 50% after 10 h of process. In Gryta's research group, it was  
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41 497 confirmed that ethanol, together with volatile substances, can be removed from fermentation  
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43 498 broths with MD (Gryta & Barancewicz, 2011). In this case, they noticed that acetic acid and  
44  
45 499 propionic acid were also carried and evaporated from the feed to distillate.

46  
47 500 In a recent study, Kumar et al. (2017) experimented bioethanol production integrating MF, NF  
48  
49 501 and DCMD unit operations, as observed in **Figure 5**. This planned strategy was able to operate  
50  
51 502 for several hours with no signal of concentration polarization effect and flux decay. Here,

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3 503 continuous ethanol production from sugarcane juice has been successfully done with high  
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5 504 productivity (ca.  $9.2 \text{ g L}^{-1} \text{ h}^{-1}$ ) and a yield  $0.47 \text{ g g}^{-1}$ . In this work, the DCMD unit acted as  
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7 505 final stage, improving the product purity and concentration enrichment. As an author's  
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9 506 suggestion, the integrated membrane system can be operated using solar energy, which can  
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11 507 make the process more energy efficient and eco-friendly for ethanol purification.  
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509  
510 **Figure 5.** Integration of micro, nanofiltration and membrane distillation for the continuous production  
511 of bioethanol. Adapted from (Kumar et al., 2017).  
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3 513 [Banat & Al-Shannag \(2000\)](#), for instance, studied MD for the recovery of diluted acetone-  
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5 514 butanol-ethanol in aqueous solution. According to the outcomes reported, the effectiveness of  
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7 515 MD process in compensate the inhibitory effect displayed by the acetone-butanol-ethanol on  
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9 516 the microbial culture. Also, the feed temperature increase contributed to increase the butanol  
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11 517 selectivity, which is indeed highly toxic solvent. Optimizing the process conditions, the optimal  
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13 518 feed temperature for butanol extraction was found to be 55 °C, independently from its high  
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15 519 boiling point in comparison with acetone and ethanol.

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19 520 In the beer manufacturing industries, there is challenge of manufacturing non-alcoholic, or low  
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21 521 alcoholic content, beverages from alcoholic beverages ([Roberto Castro-Muñoz, 2019](#)). For this  
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23 522 task, it is pursued to remove the ethanol once the fermentation in the beer is achieved. Hence,  
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25 523 [Purwasasmita et al. \(2015\)](#) attempted beer dealcoholization by proposing VMD. The authors  
26  
27 524 basically studied the effect of pressure of both feed and permeate streams on the flux and  
28  
29 525 selectivity. For the process, a non-porous thin-film composite polyamide (commercial known  
30  
31 526 as TW30-1812-75 from DOW Filmtec from Dow Chemical Company) was equipped.  
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33 527 Interestingly, VMD was able to reduce the alcohol content, from 5 to 2.45%-vol., operating  
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35 528 within first 6 h. According to author's report, there was no loss of any nutrient and flavoring  
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37 529 substances; however, a minimal loss of maltose was observed attributed to the possible  
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39 530 adsorption on the membrane surface.  
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#### 4. Conclusions and perspectives in the field

46  
47 532 MD has been successfully applied in processing of foods and food bioactive substances thanks  
48  
49 533 to its lower energy requirement and easy operation compared with traditional distillation. This  
50  
51 534 membrane processes substantially contributes to the preservation of thermal sensitive  
52  
53 535 bioactives molecules, resulting in high quality of products. The main application in food  
54  
55 536 industry deal with the concentration of fruit juices and natural extracts, ethanol removal from  
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3 538 alcoholic beverages, processing of dairy products, as well as the recovery and concentration of  
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5 539 bioactive substances from food products and wastes. However, this review identifies that most  
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8 540 of the applications have been done at lab scale, but promisingly, MD has initiated to be applied  
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10 541 in pilot and large scale developments (Sotoft et al., 2012).

11  
12 542 As for juice concentration, MD will continue being explored in near future thanks to the high  
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14 543 productivities in terms of permeate flu, ranged from 0.6 to 28 L m<sup>-2</sup>h<sup>-1</sup> (see **Table 1**). In this  
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16  
17 544 latter application, it is likely that PVDF is the most used polymer material for the fabrication of  
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19 545 MD with hydrophobic properties. Current scopes of research are still emphasized on improving  
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21 546 the properties of this polymer, as well as the resulting properties when using new strategies and  
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23  
24 547 items (green solvents) for membrane fabrication (Zou, Hu, et al., 2022; Zou, Kim, et al., 2022).  
25  
26 548 In addition to this last point, a new trend deals with the evaluation of new hydrophobic materials  
27  
28 549 focused on emerging fabrication protocols and hybrid materials, e.g., such as flat sheet  
29  
30 550 electrospun based on PSF/zeolite (Estedlali et al., 2021).

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33 551 Specific configuration of MD set-ups, such as VMD, are capable to recover aroma substances  
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35 552 at the early stages of juice processing (Sotoft et al., 2012). Here, the research on membrane  
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37 553 material and properties will be crucial for the control of wetting and fouling in VMD long-term  
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39  
40 554 applications.

41  
42 555 Over the reviewed applications, the flux decline over time as a consequence of membrane  
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44 556 fouling is still one of the main challenges for food processing via MD. To control the membrane  
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47 557 fouling, several suggestions can be given, as follows: control the physicochemical properties of  
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49 558 the feed, improve the hydrophilic properties of the membranes, or integrate the pre-clarification  
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51 559 process prior to MD being applied. Of course, the prominent fouling and wetting of membrane  
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53 560 pores negatively influence the membrane performance and durability, limiting the application  
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56 561 of MD in this field. For this, research on long-term MD performance is required.

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