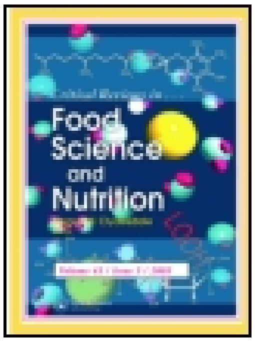

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Membrane-based technologies for meeting the recovery of biologically active compounds from foods and their by-products

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Abstract

To date, according to the latest literature inputs, membranes-based technologies (microfiltration, ultrafiltration and nanofiltration) have demonstrated to meet the recovery of biologically active compounds, mainly phenolic compounds and their derivatives, from agro-food products and by-products. The goal of this paper is to provide a critical overview of the on ongoing development works aimed at improving the separation, fractionation and concentration of phenolic compounds and their derivatives from their original sources. The literature data are analyzed and discussed in relation to separation processes, molecule properties, membrane characteristics and key factors affecting the performance of such technologies. Technological advances and improvements over conventional technologies, as well as critical aspects to be further investigated are highlighted and discussed. Finally, a critical outlook about the current status for a large-scale application and the role of these processes from an environmental viewpoint is provided.

Keywords: *phenolic compounds; foods; agro-food by-products; ultrafiltration, nanofiltration.*

Nomenclature

MF: Microfiltration

UF: Ultrafiltration

NF: Nanofiltration

MWCO: Molecular weight cut-off

OMW: olive mill wastewater

1. Introduction: Background of membrane-based technologies for the processing of foods and agro-food by-products

Today, the use of membrane-based technologies, such as micro- (MF), ultra- (UF) and nano- (NF) filtration, is potentially considered for several applications in the food processing industry. Since the last two decades these technologies have been used for clarification of natural products (e.g. natural extracts, fruit juices, etc.) (Castro-Muñoz et al., 2017a; Destani et al., 2013), while in this last decade they have extensively proposed for the treatment of agro-food by-products (wastewaters, food wastes, etc.) in order to reduce their polluting load and, consequently, for the final waste disposal. In recent years, however, increasing attention has been devoted to the possibility of valorising agro-food residues through the separation and recovery of high-added value compounds. Thanks to their intrinsic properties, membrane-based processes are an emerging tool to improve the currently adopted valorisation protocols, within a sustainable biorefinery strategy, with remarkable improvements of the environmental and economical sustainability of the overall approach (Castro-Muñoz et al., 2018a; Galanakis, 2013). Over the last decades, the implementation of pressure-driven membrane operations in agro-food productions has increased remarkably, as evidenced by the number of scientific publications in this field (**Figure 1)**. Indeed, membrane processes offer several advantages over conventional separation methodologies, including mild operating conditions of temperature and pressure, therefore preserving the functional properties of food products, non-use of chemical agents or solvents, and, consequently, product contamination. In addition, they are characterized by high selectivity towards specific compounds, simple equipment, relatively easy scale-up, reduced number of processing steps and low energy consumption (Conidi et al., 2014a).

Figure 1. Evolution of the use of membrane-based technologies for the processing of food and by-products (by Scopus, April 28th, 2018).

In the light of these properties and the continuous demand of both manufacturers and consumers for minimally-processed foods free of external contaminants and health-promoting foods, the recovery of natural antioxidants by membrane-based operations has been largely investigated in the last years (Cassano et al., 2016a; Galanakis et al., 2016; Martín et al., 2018; Castro-Muñoz et al., 2018b) with particular interest towards phenolic compounds and their derivatives (Castro-Muñoz et al., 2016a). Practically, the recovery lies the split of a feed bulk solution (e.g. extract, fruit juice, wastewater, by-product) by means of a perm-selective porous barrier well called “membrane”, under a transmembrane pressure applied between the two side of the membrane: this leads to the partial fractionation of the feed solution into two new streams: a permeate stream consisting of the solvent (commonly water) that passes through the membrane and solutes with lower molecular weight than the membrane's nominal molecular weight cut-off (MWCO), and a retentate stream containing all compounds partially or totally retained (higher molecular weight compounds) by the membrane (**Figure 2**).

Figure 2. General drawing of pressure-driven membrane technology.

The valorization of food manufacturing waste is actually one of the current challenges for scientists (Mirabella et al., 2014). Industrial ecology concepts, like cradle-to-cradle and circular economy, are considered as leading principles for eco-innovation, aiming at “zero waste economy” where the wastes are used as raw material for new products and applications. In this sense, there is a strong interest in encouraging the implementation of the integral strategy “5-Stages

Universal Recovery Process” (Galanakis, 2012; Galanakis, 2015a), which involves the partial and/or complete recovery of compounds from food wastes through sequential steps as: (i) macroscopic pre-treatment, (ii) separation of macro- and micromolecules, (iii) extraction, (iv) isolation-purification and (v) product formation. When dealing with membrane-based technologies this fifth stage cannot be easily reached, but whatever of the others can be met surely. Particularly, UF and NF have been recognized for their capability to recover bioactive compounds from agro-food processing wastes, also through their combination in hybrid processes (Cassano et al., 2018; Dhillon et al., 2013). According to literature data obtained mainly on laboratory scale, the performance of these processes is affected by several parameters that should be carefully evaluated and optimized case-by-case for a real application and scale-up. Thereby, all these aspects are addressed and discussed in detail according to the latest literature inputs. In addition, the role of these processes on environmental viewpoint is provided.

2. Key factors influencing the performance of membrane processes

The performance of MF, UF and NF processes, in terms of solute rejection and permeate fluxes, depends on a number of factors such as (Fane and Fell, 1987; Cassano et al. 2018; Astudillo-Castro, 2015):

i) Physico-chemical composition of the feedstream: this is a critical parameter having a strong influence on membrane fouling. Basically, membrane fouling is the main phenomenon that produces a decline in permeate flux with time of operation (Fane and Fell, 1987). In general, the term is used to describe a long term flux decline caused by accumulation of certain materials at the membrane surface. It may occur due to a concentration polarization layer development over the membrane surface, the formation of a cake layer and/or a blockage of the membrane pores. Membrane fouling is the result of specific interactions between the membrane and various solutes in the feedstream. Therefore, the physicochemical characteristics of individual feed components (i.e. conformation, charge, hydrophobic interactions, zeta potential, etc.) have a significant impact on these interactions.

For example, phenolic compounds have demonstrated their adsorption on polyethersulfone MF membranes which take place due to polar interactions (Susanto et al. 2009; Cartalade and Vernhet, 2006). In addition, phenolic compounds may interact together and with other compounds (i.e. polysaccharides) to form large particles which have a negative impact during filtration process. Therefore, although these compounds are much smaller than the average pore size of MF membranes, their interactions and those with membrane surface and membrane pores play a key role in membrane fouling (Vernhet and Moutounet, 2002).

ii) Operating parameters:

Process engineering factors, such as transmembrane pressure, cross-flow velocity and temperature have a strong influence on membrane fouling, and consequently, on membrane productivity and selectivity. For instance, the temperature tends to increase the permeate fluxes in porous membranes. An increase in temperature results in a decrease in the viscosity of the fluid, which reduces the resistance to flow and promotes turbulence. In addition, the increase in temperature also increases the diffusivity and hence the rate of transport of solutes carried away from the membrane surface and back into the bulk stream (Ramli and Bolong, 2016). Regarding the transmembrane pressure, there is a linear relationship between the pressure and the permeate flux, which is valid until reach the limiting transmembrane pressure. At this limiting pressure or higher, the permeate flux is not a function of pressure; basically, the permeation is governed by the fouling and concentration polarization phenomena (Astudillo-Castro, 2015). On the other hand, the rejection of some compounds (e.g. phenolic compounds) usually increases by increasing the transmembrane pressure (Conidi et al. 2011; Díaz-Reinoso et al. 2009). This is attributed to the formation of a thin layer near the membrane surface, which represents an extra barrier, and thus increase the retention coefficients (Bacchin et al., 2002). Moreover, when pressure increases also promote the membrane fouling that acts as an additional resistance (Castro-Muñoz et al. 2016b).

Higher flowrates tend to remove deposited materials and, consequently, reduce the hydraulic resistance of the fouling layer. However, in some cases the increasing of the cross flow velocity determines a removal of greater particles from the membrane surface and a stratification of smaller particles on the membrane surface with a consequent pore plugging.

iii) Membrane features: Membrane properties, including hydrophilicity/hydrophobicity, surface topography, charge and pore size have a strong influence on membrane-solute interactions, and hence on membrane fouling phenomena.

The pore size is the main characteristic that distinguishes MF, UF and NF membranes, playing a key role in the separation process. Typical pore-size, operating pressure requirements and separation mechanisms of MF, UF and NF membranes are reported in **Table 1**.

Table 1. Features of MF, UF and NF (adapted from Cassano et al., 2018; Castro-Muñoz et al., 2016a)

In theory, the ability of pressure-driven membrane processes to separate components from a fluid stream is based on a molecular sieving mechanism according to their molecular weight; however, membranes' MWCO is not the only parameter. For instance, the asymmetric characteristics of the membrane pores do not always reflect a narrow MWCO range (Galanakis, 2015b). This is could be attributed to the preparation procedure of the membranes; for instance, the wet phase inversion, as the most used membrane preparation method for UF and NF membranes, generally leads to obtain asymmetric porous membranes (Blanco et al., 2006; Boussu et al., 2006). Typically, it has been well documented that at when the polymer solution comes into contact with the non-solvent in the coagulation bath, a rapid outflow of the solvent from the casting solution to the coagulation bath causes higher-concentration polymer molecules to be aggregated at the top layer, well-known as “thin or skin layer”, which basically contributes to increase the solute rejection rates. **Figure 3** depicts clearly the typical view of an asymmetric polysulfone UF membrane (Ali et al., 2011).

Figure 3. Cross section view of an asymmetric UF membrane (Ali et al., 2011).

UF and NF membranes are usually prepared with an additional layer in order to provide higher rejection efficiencies: this is performed by depositing a top layer on the membrane’ surfaces. This approach has been commercially consolidated. **Table 2** shows some characteristics of commercial UF and NF composite membranes.

Table 2. Characteristics of some commercial UF and NF membranes.

The surface roughness also plays an important role in the performance of UF and NF membranes. It is well known that membrane fouling increases on rougher surfaces (Evans et al., 2008). For example, the presence of protuberances on the surface of polyamide-based membranes seems to be responsible of membrane fouling due to a suspended matter capture. On the contrary, cellulose acetate membranes exhibit smoother surfaces and are less prone to fouling.

Most membranes exhibit a net negative charge under usual process conditions; consequently, electrostatic forces can be involved if solutions containing charged particles are processed. The surface charge on the membrane depends on type of membrane material, but the pH and ionic strength of the feed solution also contribute (Galanakis et al., 2002). This surface charge becomes important if charged molecules, such as proteins, are being processed (Kanani, 2015). On the other hand, hydrophobicity of membrane material is crucial as well; for instance, hydrophilic membranes tend to give better performance when using aqueous feedstreams (Kanani, 2015). On the other hand, Coulombic and hydrophobic interactions between the solutes and membrane surface (phenolics-membrane, phenolics-phenolics) can contribute to the solute rejection (Crespo and Brazinha, 2010; Cassano et al., 2017). Based on all aforementioned characteristics, tight UF membranes (1-3 kDa) are considered as the border of NF, displaying high separation performance in concentrating low molecular weight compounds (e.g. anthocyanins, low molecular weight polyphenols, low molecular weight sugars, and peptides). Thereby, the following section provides an overview of the biological compounds that can be recovered by UF and NF membranes, paying particular attention in relevant experimental studies.

3. Biologically active compounds recovered by means of UF and NF membranes

3.1. Foods

The clarification of several types of natural extracts like fruit and vegetable juices is the most developed application of membrane operations in food processing. This technology has emerged as a valid alternative to the traditional juice clarification due to its advantages over traditional clarification methodologies (i.e. use of adsorbents, gelatins and other filtration aids) in terms of less manpower requirement, reduced operating costs and working times, increased yields, possibility of operating in a single step, easy cleaning and maintenance of the equipment, reduction of waste products and elimination of needs for pasteurization. In addition, possibility to operate at low temperature contributes to maintain the organoleptic properties of the juices (Bhattacharjee et al., 2017). UF and MF have been successfully used for processing of fruit and vegetable juices including lemon (Chornomaz et al., 2013), tomato (Razi et al., 2012), pomegranate (Conidi et al., 2017), cactus pear (Castro-Muñoz et al., 2015), bergamot (Conidi et al., 2011), grape (Cancino-Madariaga et al. 2012), blood orange (Cassano et al., 2007a), passion fruit (De Oliveira et al., 2012), mandarin (Cassano et al., 2009), apple (Echavarría et al., 2012), pineapple (Laorko et al., 2010) and kiwi (Cassano et al., 2004), to mention just a few. The clarification step deals with the removal of high molecular weight compounds including suspended solids, colloids and gel-forming agents that provide turbidity, while small solutes such as vitamins, salts and sugars flow together with water. This process allows to obtain a clarified fraction (permeate) free of spoilage microorganisms and a fibrous concentrated pulp (retentate). Basically MF is the most used technology for juice clarification at industrial level. Moreover, the fruit juice and beverage industry also use UF for both clarification and concentration depending on membranes' MWCO; but more finely, UF is used to clear up a wide assortment of natural juices by evacuating polluting components (yeast, molds, micro-organisms, colloids, proteins, tannins, and polysaccharides), conferring stability to the last item (Ilame and Singh, 2015).

MF or UF can result in different efficiency levels in reducing juice clarity and in the recovery of biologically active compounds. Cassano et al. (2010) evaluated the physicochemical composition of cactus pear juice clarified by using both MF and UF membranes. The clarified juice resulted enriched of antioxidant compounds (polyphenols, Vitamin C, etc.), sugars, amino acids and minerals independently by the selected membranes. On the other hand, Mirsaeedghazi et al. (2012) used the above techniques to remove antioxidant compounds from pomegranate juice. According to their results, there was a greater membrane fouling in the UF process when compared to the MF process. MF was also used as pre-treatment step of the juice to reduce membrane fouling of UF membranes. The UF of microfiltered pomegranate juice did not have any influence on the turbidity but decreased the levels of valuable components, such as total anthocyanin and antioxidant activity levels.

NF membranes are mainly used to concentrate fruit juices, to regulate sugar concentration (García-Martín et al., 2010) and to separate phenolic compounds from sugars (Wei et al., 2007; Conidi et al., 2017). **Table 3** summarizes typical bioactive compounds recovered by MF, UF and NF membranes from fruit and vegetable juices.

Table 3. Biologically active compounds recovered from fruit and vegetable juices using membrane-based technologies.

Generally, these processes offer good recovery rates for several bioactive compounds; for example, MF technology is able to recover from 47 up to ~100% of anthocyanins, glutamine, isoproline, proline, betanin, isobetanin, sugars, galacturonic acid and some phenolic compounds, all these in permeate streams. While UF technology, depending of its membranes' MWCO, can provide recovery rates between 44-99% of those compounds, which can be mainly contained in permeate; however, some of these compounds can start to be rejected by the membranes (commonly through tight UF), and thus partially recovered in the retentate. NF technology leads to pass primarily water: this allows to concentrate bioactive compounds in retentate in the range from 50 up to 99%.

In recent years the interest of researchers and consumers for the development of functional ingredients from pomegranate or its derivative products has increased significantly. Indeed, clinical and biological research studies have confirmed the fruit's health-beneficial effects mainly attributed to its phenolic fraction containing a significantly high level of hydrolyzable tannins, as well as anthocyanins (delphinidin, cyanidin and pelargonidin 3-glucosides and 3,5-diglucosides), which exhibit high antioxidant activity (Kalaycıoğlu and Erim, 2017).

Accordingly, clarification, extraction and purification procedures based on the use of membrane operations have been investigated as alternative to conventional methodologies in order to preserve bioactive compounds of the juice and to develop innovative formulations for the development of functional foods meeting the consumer requirements (Baklouti et al., 2012; Cassano et al., 2015a; Conidi et al., 2017).

Cassano et al. (2015b) evaluated the performance of modified poly(ether ether ketone) (PEEKWC) and polysulfone (PS) hollow fibre membranes, prepared through the phase inversion process, in the clarification of pomegranate juice with the aim to preserve the phenolic fraction and to obtain high permeation fluxes. The prepared membranes had a similar MWCO: 2.0 kDa for PS membranes and 2.5 kDa for PEEKWC membranes. PS membranes exhibited higher productivity when compared with PEEKWC membranes in the treatment of the juice according to a batch concentration configuration. In addition, these membranes showed lower rejections towards flavonoids and total phenols when compared with PEEKWC membranes. For PS membranes maximum permeation fluxes were obtained at an operating pressure of 0.6 bar, a temperature of 25 °C and an axial feed velocity of more than 4 ms^{-1}. Overall, experimental results indicated that the membrane material plays a key role in the separation of bioactive compounds from the juice.

Recently, UF and NF membranes with MWCO ranging from 1,000 to 4,000 Da have been tested to purify bioactive compounds from clarified pomegranate juice (Conidi et al., 2017). Among the selected membranes, a thin-film composite membrane with a MWCO of 2,000 Da (Desal GK, from GE Osmonics), displayed higher permeate fluxes, lower fouling index and a good separation efficiency of sugars from phenolic compounds in comparison with the other tested membranes. Retentate and permeate fractions enriched in phenolic compounds and sugars, respectively, were obtained through concentration/diafiltration experiments in selected operating conditions. Yields of polyphenols and anthocyanins in the retentate stream were of the order of 84.8% and 90.7%, respectively. The retentate fraction, characterized by a high antioxidant activity, was considered of interest for the formulation of nutraceutical products or as a natural colorant in alternative to the use of synthetic ones; on the other hand, the permeate and diafiltrate streams, enriched in sugar compounds, were proposed to be reused as food additives or as bases for soft drinks. The proposed process scheme for the fractionation of pomegranate juice and the related mass balance of phenolic compounds and anthocyanins is illustrated in **Figure 4**.

Figure 4. Integrated membrane process proposed for the recovery of phenolic compounds and anthocyanins from pomegranate juice (adapted from Conidi et al., 2017).

3.2. Agro-food processing by-products

It is well known that the activities of food processing industries are characterized by the production of large amounts of by-products and waste whose management creates serious problems, both from the economic and environmental point of view. On the other hand, many of these residues are a source of useful compounds which can be potentially reused into other production systems, through e.g. bio-refineries and the development of new products with a market value (i.e. functional foods) (Mirabella et al., 2014). Among these compounds, polyphenols have been deeply studied for their great protective activities as antioxidants and their contribution to the prevention of several types of human diseases, such as cancer, cardiovascular and neurodegenerative diseases (Galanakis et al., 2015; Visioli et al., 2011). Several agro-food by-products, including wastewaters (from olive, artichoke, maize, and winemaking processing industries), residues (orange press liquor, winery effluent, fruit seeds, fermented grape pomace, grape marc, etc.) and some other derivative by-products represent a potential source of phenolic compounds (Cassano et al. 2016a; Cassano et al. 2018; Castro-Muñoz et al. 2016b). These agro-residues are characterized by a complex physicochemical composition; therefore, the recovery of phenolic compounds from these sources require several steps (i.e. coagulation and precipitation of impurities, adsorption on resins) based on the use of chemicals, solvents and high temperatures which account for a significant part of the total costs of polyphenol production.

In the light of their intrinsic properties and related advantages over conventional separation methodologies, membrane processes have been largely investigated in the last years for the recovery of phenolic compounds from agro-food processing residues.

Table 4 summarizes the main phenolic compounds recovered from different agro-food by-products by means of membrane-based technologies.

Table 4. Phenolic compounds recovered from agro-food by-products using membrane-based technologies.

Among these by-products, wastewaters arising from the olive oil extraction process, named as olive mill wastewaters (OMWs), have been largely investigated.

In the Mediterranean countries, the olive oil production represents as one among the oldest agro-food industries, with a production of about $1.8x10^6$ ton olive oil per year. This leads to produce over 30 million m^3 OMWs per year (Haddad et al., 2017).

OMWs are effluents with an intensive violet-dark brown color and strong specific olive oil smell constituted in average by 83.4% of water, 1.8% of inorganic salts and 14.8% of organic compounds. Organic substances determine the high polluting load of these wastewaters: biochemical oxygen demand (BOD5) and Chemical Oxygen Demand (COD) may be as high as 100 and 200 g/L, respectively. The organic fraction contains sugars, tannins, polyphenols, polyalcohols, pectins, lipids, proteins and organic acids (Dermeche et al., 2013).

Today, the integrated management of this by-product has been pointed out as a real challenge by the olive professional sector and environment protection and preservation agencies. In this framework, many approaches have been proposed for its treatment and exploitation such as composting, flocculation, chemical treatments, agricultural water source, a feedstock for biofertilizers production and conventional centrifugation-filtration operations (Haddad et al. 2017; Roig et al. 2006); however, all these approaches lack the recovery of high-added value compounds totally or partially dissolved in the OMWs.

Pressure-driven membrane operations have gained a great attention as promising technologies for the valorization of OMWs. The treatment of OMWs by membrane operations is generally focused on the development of integrated systems for the recovery, purification and concentration of polyphenols from OMWWs with regard to their specific MWCO values and the production of effluents of acceptable quality for safe disposal into the environment.

It is notable mentioning that tight UF and NF membranes are able to recover specific derivative phenolic compounds, such as hydroxytyrosol, catechol, tyrosol, caffeic and p-cumaric acids, displaying over 80% recovery rate. A conceptual process design for the recovery of phenolic compounds from OMWs was proposed by Cassano et al. (2013) on the basis of experimental results obtained on laboratory scale. According to this process, a first UF step performed with hollow fiber membranes with a nominal pore size of 0.02 μm (HSF, Toray) allowed to remove suspended solids from the raw wastewater. The UF permeate was then processed with a flat-sheet UF membrane (Etna 01PP, Alfa Laval) with a MWCO of 1000 Da. Both UF membranes showed low rejections towards low molecular weight polyphenols (2.1 and 17.6% respectively). The UF permeate form the second UF step was then concentrated by NF by using a spiral-wound membrane (NF90, Filmtec/Dow) with a salt rejection higher than 97% (MWCO of about 200 Da). The NF membrane retained all the analyzed phenolic compounds producing a permeate depleted in phenolic compounds. The proposed process, illustrated in **Figure 5**, allowed to obtain a purified fraction containing more than 85 mg/L of low molecular weight polyphenols (including hydroxytyrosol, protocatechuic acid, catechol, tyrosol, caffeic acid, and p-cumaric acid) displaying high antioxidant activity (2,175 mg L^{-1} Trolox). This fraction was considered of interest for cosmetic, food and pharmaceutical industries as liquid, frozen, dried or lyophilized formulations. The NF permeate, depleted in phenolic compounds, was suggested to be reused in the olive oil extraction process as process water or as membrane cleaning solution.

Figure 5. Integrated membrane process proposed for the recovery of phenolic compounds from OMW (adapted from Cassano et al., 2013).

Integrated membrane processes for the recovery of phenolic compounds from artichoke wastewaters has been also recently investigated (Cassano et al. 2015a; Conidi et al. 2014a). Basically, raw wastewaters are pre-treated by UF membranes (MWCO 50 kDa) in order to remove inulin and suspended solids; the UF permeate is submitted to a first NF process by using a polyethersulphone spiral-wound membrane module (NP030, Microdyn Nadir) with a MWCO of 400 Da. This step allows to produce a retentate fraction enriched in phenolic compounds suitable for nutraceutical, cosmeceutical or food application. The NF permeate is processed through cross-linked aromatic polyamide membranes with a MWCO of 150–300 Da (i.e. Desal DL ad DK, GE Water & Process Technologies) producing a retentate fraction enriched in sugar compounds and a clear permeate which can be reused as process water or for membrane cleaning.

Target compounds such as chlorogenic acid, apigenin and cynarin were highly retained by the NP030 membrane (rejections were more than 82%). An increased concentration of these phenolic compounds in the NF retentate, accompanied by an increasing of the total antioxidant activity, was observed by increasing the volume reduction factor of the process.

Integrated membrane processes aimed at separating sugar compounds from phenolic compounds have been also proposed for by-products of citrus processing industry such as orange press liquor and bergamot juice (Cassano et al. 2014; Conidi and Cassano 2015). Feedstreams were pretreated by UF in order to remove suspended solids and macromolecular compounds. The UF permeate was then treated by NF in order to produce retentate fractions enriched in phenolic compounds. In this approach polyethersulphone membranes with MWCO of 1,000 Da (i.e. NP010, Microdyn Nadir) showed the largest gap between the rejection coefficients towards sugar and phenolic compounds.

The concept of integrated membrane design was also recently applied for the valorization of the primary by-product produced by the Nixtamalization process of maize, named as Nixtamalization wastewaters and well known as “Nejayote” (Castro-Muñoz and Yañez-Fernandez, 2015; Castro-Muñoz et al., 2016b). This typical maize pre-treatment is generally carried out in America, producing a huge amount of wastewaters: in particular, the estimated volume generated in Mexico is around 14.4 million m^3/ year (Castro-Muñoz et al., 2017b). This wastewater contains significant amounts of soluble and insoluble solids consisting of organic and inorganic compounds including bioactive compounds such as carbohydrates, dietary fibers and polyphenols (Gutiérrez-Uribe et al., 2010).

Today, researchers are trying to face the overproduction of this extract through its integral management. To date, different uses have been proposed, e.g., for animal feeding stock, removal of its chemical oxygen demand through bioreactors and coagulation-flocculation treatments, microorganisms' growth medium, production of food additives and food technological approaches (Acosta-Estrada et al., 2014; Castro-Muñoz et al., 2017b; Valderrama-Bravo et al., 2012).

The potential of an integrated membrane process based on the use of MF and UF membranes for the recovery of bioactive compounds from Nejayote has been recently investigated by Castro-Muñoz and Yañez-Fernandez (2015). Raw wastewaters were treated by PS hollow fiber MF membranes (CFP-1-E-4A, Amersham Biosciences Corp.) with nominal pore size of 0.2 μm in order to remove suspended solids and high molecular weight compounds. This step allowed also to reduce fouling phenomenon in the next UF process. The latter was performed by using two different PS hollow fiber membranes (UFP-100-E-4A and UFP-1-E-4A, Amersham Biosciences Corp.) with MWCO of 100 kDa and 1 kDa, respectively (**Figure 6**).

Figure 6. Integrated membrane process proposed for the recovery of phenolic compounds from NWs (adapted from Castro-Muñoz and Yañez-Fernandez, 2015).

This sequential design let to recover about 45% of the initial polyphenols content in the permeate of the final UF step, corresponding to a final content of phenolic compounds of 950 mg L^{-1}. This fraction presented an antioxidant activity of 1.56 μmol Trolox mL^{-1}

(Castro-Muñoz et al., 2016b). It is worth mentioning that most part of the total calcium hydroxide (about 82%) was removed from the extract through the UF 1 kDa membrane, while the overall integrated process reduced around 81% of the total organic carbon contained in the extract. Finally, it is likely that the polyphenols recovery rate from Nixtamalization wastewaters could be enhanced by using NF membranes, which can meet the concentration of target compounds in retentate streams.

Wastewaters ad by-products of the winemaking industry represent another inexpensive source of beneficial phytochemicals, which could be successfully used in food, cosmetic and pharmaceutical industries. The combination of membranes with different MWCO to fractionate phenolics (gallic acid, catechin, gallates, etc.) from defatted milled grape seeds extracts was investigated by Santamaría et al. (2002). A fractionation sequence based on the use of UF and NF membranes was developed for purification of fractions of proanthocyanidins with different MW.

An integrated membrane process for the fractionation of winery effluents generated in the second racking was proposed by Giacobbo et al. (2017a). The proposed design is based on the fractionation of a microfiltered effluent through a sequence of UF and NF membranes with decreasing MWCO. UF membranes with MWCO of 1 and 10 kDa (Etna 01PP and 10PP, from Alfa-Laval) allowed to recover most part of phenolic compounds in the permete stream while rejecting polysaccharides. The UF permeate was concentrated by NF membranes with MWCO of 150-300 Da (NF270, Dow/Filmtec) having a full rejection to anthocyanins and lower rejections to total organic carbon.

The proposed process, illustrated in **Figure 7**, allows to produce: (i) a concentrated UF fraction enriched in polysaccharides; (ii) a concentrated NF fraction enriched in bioactive compounds, with potential applications in the pharmaceutical, cosmetic and food industries; (iii) a NF permeate stream reusable as process water.

Figure 7. Integrated membrane process for the fractionation of wine lees (adapted from Giacobbo et al., 2017a).

The valorization of cereal by-products is another topic of great interest associated to both economic and environmental aspects (ElMekawy et al., 2013). In particular, oat (*Avena sativa*) and oat by-products have been proven to be helpful in the treatment of diabetes, cholesterol and cardiovascular disorders (De Schrijver et al., 1992; Tapola et al., 2005).

The recovery of β-glucan from oat mill waste by UF was investigated by Patsioura et al. (2011). Polyethersulphone membranes with a MWCO of 100 kDa exhibited higher permeate fluxes and lower retention coefficients towards β-glucan (<81%) when compared with regenerated cellulose and polysulphone membranes of similar MWCO.

The optimized UF process allowed to recover β-glucan in the retentate stream, while smaller organic and inorganic compounds were recovered in the permeate stream (membrane retention was lower than 15%).

Polysulphone UF membranes of 100 kDa allowed also to recover proteins from Halloumi cheese whey, a by-product of traditional cheese of Cyprus, with a negligible retention (2-7%) towards non-reducing sugars (mainly lactose). A combination of 20 kDa-polysulphone and 2 kDa-polyethersulphone membranes produced higher recovery of proteins (87-90%) and retention of non-reducing sugars of 32-39% (Galanakis et al., 2014).

4. Weakness of membrane-based technologies

As reported previously, in the selected agro-food applications membrane processes compete directly with conventional separation methodologies due to their intrinsic advantages. In addition, the combination of membrane unit operations in integrated membrane systems or with conventional methodologies results in reliable and cost-effective treatment combined with high product quality and reduced energy consumption. However, some critical aspects have to be carefully considered in the application and implementation of membrane-based processes in specific industrial sector including the agro-food ones. When dealing with the weakness of these membrane-based technologies inherently deals with their disadvantages, which can be highlighted as, ***i) Purity restrictions:*** membrane processes rarely produce two pure streams; it means, any of the streams always contains a minor amount of an undesired component. For example, the permeate stream can present a significant amount of materials which is trying to concentrate in the retentate because the membrane selectivity is not infinite. At this point, it is important to mention that the storage and use of the membranes plays an important role, e.g. if the membrane receives suitable cleaning procedures without modifying its structure can be reused as long as needed until to its shelf life. It is clear that the ***ii) fouling*** is the key issue of these technologies, which is directly associated to the physicochemical composition of feed solutions, membrane material and configuration as well as operating and fluidodynamic conditions. This fouling, especially if it is difficult to remove, can greatly restrict the permeation rate through the membranes and make them essentially unsuitable for such applications. Typical procedures to control membrane fouling involve pretreatment of the feed solutions (e.g. sedimentation-decantation, centrifugation, flocculation, enzymatic hydrolysis, screening), membrane surface modification, hydrodynamic optimization of the membrane module and membrane cleaning with proper chemical or enzymatic detergents. At this last purpose, there are many enzymatic (Ultrasil® 62 and 53, Filzym® 161, A6, A1, 10,11) and chemical (Ultrasil® 13, OptiClean™ A, Ultrasil® 10A, AMI Chemicals® AM-55) products commercially available that generally hydrolyze most of the pollutants, such as e.g. polysaccharides, proteins, polysaccharide-like, protein-like materials and humic substances (Nguyen et al., 2010). In addition, the use of integrated membrane processes can contribute to reduce the fouling in membranes; this integrated membrane process involves the implementation of multiple membrane operations in sequence, which contribute to mitigate the occurrence of fouling phenomena in the subsequent membrane steps by prepending high pore size membranes. Typical examples of mitigation of membrane fouling in UF and NF membranes have been illustrated in the fractionation of agro-food by-products such as artichoke wastewaters (Conidi et al., 2014a), artichoke brines (Cassano et al., 2016a), OMWs (Cassano et al., 2013; Russo, 2007) and cellulose alkaline by-products (Cassano et al., 2016c). The use of integrated membrane processes requires more operation steps in order to achieve high recovery rates; however, the suitable selection of the membranes and sequence design can provide a potential alternative for the fractionation of the food or by-product systems. Finally, “membranologists” are looking for new materials or enhancing the existing ones, aiming the development of smart composite membranes which can be less affected by foulants, this is attempted by handling the hydrophilicity, membrane charge, and membrane surface with a surface-bound long-chain hydrophilic molecule (Buonomenna, 2016).

iii) Thermal, mechanical and chemical limitations: Typically, membrane modules cannot operate at relatively high temperatures. This is due to the fact that most membranes are based on polymers, and most of these polymers do not maintain their physical integrity at temperatures higher than 90-100 ºC. Most polymeric are susceptible to chemical degradation by strong acid and alkaline cleaning solutions leading to a significant reduction in membrane life. In addition, some polymeric membranes have limited mechanical stability leading to a reduction in permeability at high pressures and potential membrane failure. Inorganic membranes, such as those made in alumina (Al_2O_3), titania (TiO_2), silica (SiO_2) and zirconia (ZrO_2), allow to overcome these limitations exhibiting greatly enhanced chemical, mechanical and thermal stability compared to polymeric ones. These membranes can operate at temperatures up to 500 °C, extreme pH values and can be cleaned with aggressive chemicals, organic solvents or hot water.

iv) Energy consumption-cost relationship: Pressure-driven membrane processes are considered as low energy consumption methods (Mirza 2008; Van Der Bruggen et al. 2003), which in turn can cut down the operating costs. Membrane modules represent the dominant category of direct capital costs, followed by the devices investment and their maintenance, which surely contribute significantly to overall process costs. Moreover, it may probable that the use of additional membrane operations (e.g. in integrated membrane processes) can increase the energy consumption of the overall recovery process, and thus, increase the total cost. Herein, it is important to analyze the “cost-benefit” relationship. According to Arvaniti et al. (2012) the processing cost of OMWs by membrane filtration can be covered by the exploitation of the phenolic fraction as well as of the organic fractions to be reused as fertilizers, leading to possible profit. The operational cost for the treatment of 50,000 tons of waste was estimated to be of about 1,535,740 €, that is equivalent to 30.71€ per treated m^3 of OMW. The possible profit for the same amount of waste was estimated to be 250,000€ for the nutrient fraction and 1,875,000€ for the phytotoxic fraction, whit a profit of 42.5€ per treated m^3 of waste, which allows a net profit of 11.79€ per m^3.

The entrapment of phenols from OMWs in silica particles and/or liposomes improved their water resistance revealing their suitability for the use as UV-protection booster (enhancing the ability of synthetic UV filters) in cosmetics (Galanakis et al., 2018a). Olive phenols resulted more active UV filters in a broader region of UVB and UVA compared to ascorbic acid and α-tocopherol (Galanakis et al., 2018b). Recent studies revealed also the suitability of olive phenols as antimicrobial agents in foods that undergo oxidative deterioration during cooking (i.e. bakery products) (Galanakis et al., 2018c) and as preservatives in olive and vegetable oils as well as in other foods rich in fats, e.g. meat products (Galanakis et al., 2018d). Similarly, the price of the biologically active compounds (proteins, betalains, phenolic compounds, anthocyanins and their derivatives) recovered from other agro-food sources is relatively high (e.g. ferulic acid and tocopherol prices are approximately of €113.5/100g and €225/1kg, respectively) in relation to their use in food and pharmaceutical industries, as well as their role in human health. In addition, there is a clear increasing demand in worldwide market for these products, e.g. the worldwide market for flavors and nutraceutical ingredients was estimated about € 13 billion in 2006, while more recently the US market was projected around € 5.5 billion in 2014 (food 36%, cosmetics and toiletries 27%, beverages 15%) (Brazinha and Crespo, 2014), and this demand is expected to rise 3% per year (Castro-Muñoz et al., 2018). Finally, the recovery of these high-added value compounds and its “possible” income could help to minimize the high cost derived regarding the water purification, i.e. the cost of water treatment is estimated around $1- 2 /barrel-wastewater (Lopez et al., 2018).

5. Current status for large-scale application: Environmental effects of using membrane-based technologies towards by-products

Since two decades ago, there are reports about the implementation and success of membrane processes for water treatment, where the use of a membrane step at least is included in such processes. For example, the "Minneapolis Water Works" plant (the largest potable plant in the Western Hemisphere, USA) is working through using UF operations (Pressdee and Hill, 2006). These steps have contributed to enhance significantly the quality of drinking water, providing a current capacity over 70 million gallons per day. In this sense, it is quite possible that governments will legislate to ensure the use of approaches such as those described herein in order to reduce water and environmental pollution. Membrane-based technologies are potentially better for the environment since the membrane application require the use of relatively simple and non-harmful materials (Ilame and Singh, 2015).

The full-scale implementation of membrane-based technologies for the recovery of high-added value compounds from agro-food by-products is still a critical point. Indeed, it is likely that the proof of current literature' inputs can encourage the industries to shift the role of these technologies. Furthermore, the high costs of waste disposal make it necessary for industries that use large-scale production processes to focus on waste recycling. Especially, NF seems to be the most viable technology according to its development as a purification methodology in different chemical sectors, such as production of salt from natural brine, vegetable oil processing, water softening, dairy industry (whey processing, lactose recovery, lactic acid separation), beverage industry, sugar industry (sugar beet press water, oligosaccharide filtration) (Salehi, 2014) and as a final step in industrial processes for water treatment (Torma and Csefalvay, 2018). Clearly, the implementation of membrane-based technologies for the treatment of food processing by-products is coming; this is thanks to the evidence of such processes can meet the standard parameters of drinking water, and secondarily the recovery of valuable solutes will be considered. For instance, **Table 5** shows some permeate streams obtained from the fractionation of by-products, where basically the clear permeates can be obtained having low organic loads. It is worthy to note that NF and tight UF can be able to reach such drinking water parameters.

Table 5. Permeate water streams obtained from the fractionation of agro-food by-products by integrated membrane processes (adapted from Castro-Muñoz et al., 2017a)

6. Concluding outlook

Over the course of this deep review, membrane-based technologies have been shown to be able to meet the recovery of biologically active compounds, as phenolic compounds and their derivatives, from food natural extracts and new sources such as agro-food by-products. Membrane operations such as UF and NF have clearly demonstrated their suitability in the recovery, separation and fractionation of specific phenolic compounds from different agro-food sources. On the other hand, MF membranes are useful to remove undesirable compounds from the original raw feedstreams limiting fouling phenomena in following membrane processing steps. Compared with traditional procedures, these separation technologies are economically viable not only in terms of recovery, but also because they do not require the use of other agents or/and destructive components. Thus, the recovery of high-added-value compounds from agro-food by-products is both industrially sustainable and environmentally friendly, making membrane-based technologies useful for the integral management of derivatives from food processing industries. Furthermore, the high costs of waste disposal or wastewater treatment make it necessary for industries that use large-scale production processes to focus on waste recycling. In the future, it is quite possible that governments will legislate to ensure the use of approaches such as those described herein in order to reduce water and environmental pollution.

It is likely that research and development will be focused on new implementations of NF technology as the primary tool for the recovery and concentration of phenolic compounds. Currently, the clear increasing worldwide demand towards these biologically active compounds will encourage the consolidation of membrane-based technologies.

Conflict of Interest

The authors declare no conflict of interest.

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Table 1. Features of MF, UF and NF (adapted from Cassano et al., 2018; Castro-Muñoz et al. 2016a).

Membrane process	Pore size (nm)	Required pressure (bar)		Typical separation mechanism
		Min.	Max.	
Microfiltration	100-10,000	0.1	2	Sieving
Ultrafiltration	2-100	0.1	7	Sieving
Nanofiltration	0.5-2	3	25	Sieving & charge effect

Table 2. Characteristics of some commercial UF and NF membranes.

Membrane	Desal5DL	NTR7450	NFPES10	N30F	UTC20	Desal51HL	NF-2540, NF-4040, NF-400	NF90
Manufacturer	GE Osmonics	Nitto-Denko	Nadir	Nadir	Toray	GE Osmonics	Lenntech	GE Osmonics
MWCO (Da) Sal rejection (%)	150-300	600-800	1000	400	180	150-300	98 % $MgSO_4$	99 % $MgSO_4$
Max. Temp.	90	40	95	95	35	50	45	50

(ºC)								
pH range	1-11	2-14	0-14	0-14	3-10	3-9	3-10	2-11
Type of top layer	Cross-linked aromatic polyamide	Sulfonated polyether-sulfone	Hydrophilic polyether-sulfone	Hydrophilic polyether-sulfone	Polypiperazineamide	Cross-linked aromatic polyamide	Polypiperazine amide composite	Polyamide

Table 3. Biologically active compounds recovered from fruit and vegetable juices using membrane-based technologies.

Recovered compound	*Fruit/vegetable juice*	*Recovery rate*	*Membrane technology*	*Stream recovered component*	*Membrane characteristics (MWCO/Material/ Configuration)*	*Reference*
Antioxidant compounds	Orange juice	~98.4 %	UF	Permeate	15 kDa / PVDF/ Tubular	Cassano et al., 2003
	Lemon juice	~98.4 %	UF	Permeate	15 kDa / PVDF/ Tubular	Cassano et al., 2003
	Carrot juice	~98.4 %	UF	Permeate	15 kDa / PVDF/ Tubular	Cassano et al., 2003
Ascorbic acid	Kiwifruit juice	~99.5 %	UF	Permeate	15 kDa / PVDF/ Tubular	Cassano et al., 2004
Ascorbic acid		~99.5 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Aroma compounds (Methyl butanoate, ethyl butanoate, methyl benzoate, ethyl benzoate, 3-hexen-1-ol, (E)-hexen-1-ol, 1-hexanol, 1-octen-3-ol)		N.R	UF	Permeate	15 kDa / PVDF/ Tubular	
Betacyanins	Cactus pear juice	~63.8 %	UF	Permeate	10 kDa / PSF/ Hollow fiber	Cassano et al., 2007b
Betaxanthins		~93.5 %	UF	Permeate	10 kDa / PSF/ Hollow fiber	
Ascorbic acid		62.4 %	UF	Permeate	10 kDa / PSF/ Hollow fiber	
Citric acid		62.4 %	UF	Permeate	10 kDa / PSF/ Hollow fiber	
Glutamic acid		65.5 %	UF	Permeate	10 kDa / PSF/ Hollow fiber	

Pectins	Kiwifruit juice	60.0 %	UF	Permeate	30 kDa / Cellulose/ Flat sheet	Cassano et al., 2008
Folic acid		95.6 %	UF	Permeate	30 kDa / Cellulose/ Flat sheet	
Citric acid		98.0 %	UF	Permeate	30 kDa / Cellulose/ Flat sheet	
Ascorbic acid		96.4 %	UF	Permeate	30 kDa / Cellulose/ Flat sheet	
Glutamic acid		~100.0 %	UF	Permeate	30 kDa / Cellulose/ Flat sheet	
Total polyphenols		86.4 %	UF	Permeate	30 kDa / Cellulose/ Flat sheet	
Total phenolics	Clementine mandarin juice	83.6 %	UF	Permeate	N.R/ PSF/ Hollow fiber	Cassano et al., 2009
		91.7 %	UF	Permeate	N.R./ PEEKWC/ Hollow fiber	
Flavonoids	Cactus pear juice	58.2 %	UF	Permeate	200 kDa/ PVDF/ Flat sheet	Cassano et al., 2010
Total phenolics		93.8 %	UF	Permeate	200 kDa/ PVDF/ Flat sheet	
Ascorbic acid		72.4 %	UF	Permeate	200 kDa/ PVDF/ Flat sheet	
Betacyanins		100.0 %	UF	Retentate	200 kDa/ PVDF/ Flat sheet	
Betaxanthins		77.0 %	UF	Retentate	200 kDa/ PVDF/ Flat sheet	
Proteins		100.0 %	UF	Retentate	200 kDa/ PVDF/ Flat sheet	
Ascorbic acid	Pomegranate juice	69.1 %	UF	Permeate	10% rejection dextran 68,800 MW / PEEK/ Hollow fiber	Cassano et al., 2011a
Total polyphenols (cathechin)		83.4 %	UF	Permeate	10% rejection dextran 68,800 MW / PEEK/ Hollow fiber	
Malic acid		95.7 %	UF	Permeate	10% rejection dextran 68,800 MW / PEEK/ Hollow fiber	
Citric acid		98.6 %	UF	Permeate	10% rejection dextran 68,800 MW / PEEK/ Hollow fiber	
Betalains	Purple cactus pear juice	100.0 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	Castro-Muñoz et al., 2015
Anthocyanins	Blue corn extract	~78.0 %	UF	Permeate	5 kDa / Regenerated cellulose/ Spiral wound	Cerón-Montes et al., 2015
Hesperidin	Lemon juice	~65.8 %	UF	Permeate	N.R./ PVDF/ Flat sheet	Chornomaz et al., 2013
Anthocyanins	Roselle extract	~100.0 %	UF	Retentate	5 kDa/ PES/ Flat sheet	Cisse et al. (2011a)
		~90.0 %	UF	Retentate	1 kDa/ Composite polyamide/ Flat sheet	
		~90.0 %	UF	Retentate	2 kDa/ Thin film/ Flat sheet	
		~80.0 %	UF	Retentate	20 kDa/ PES/ Flat sheet	
		~60.0 %	UF	Retentate	50 kDa/ PES/ Flat sheet	
		~90.0 %	NF	Retentate	N.R./ Composite/ Flat sheet	
		~100.0 %	NF	Retentate	0.2-0.4 kDa/ Polyamide thin-film composite/ Flat sheet	
		~100.0 %	NF	Retentate	N.R./ PES/ Flat sheet	
		~100.0 %	NF	Retentate	0.15-0.3 kDa/ Polyamide PSF thin-film / Flat sheet	
		~100.0 %	NF	Retentate	N.R./ Cross linked polyamide composite / Flat sheet	
		~100.0 %	NF	Retentate	N.R./ Composite/ Flat sheet	
Vitamin C	Roselle extract	~95.0 %	MF	Permeate	0.2 µm/ Ceramic /Tubular	Cisse et al. (2011b)
Anthocyanins		~98.4 %	MF	Permeate	0.2 µm/Ceramic/Tubular	

Total polyphenols	Bergamot juice	93.7 %	UF	Permeate	100 kDa / PSF / Hollow fiber	Conidi et al., 2011
narirutin		99.2 %	NF	Retentate	450 Da / TiO_2 / Monotubular	
naringin		95.3 %	NF	Retentate	450 Da / TiO_2 / Monotubular	
Hesperidin		91.7 %	NF	Retentate	450 Da / TiO_2 / Monotubular	
Neohesperidin		96.3 %	NF	Retentate	450 Da / TiO_2 / Monotubular	
Malic acid		99.2 %	UF	Permeate	100 kDa / PSF / Hollow fiber	
Ascorbic acid		87.5 %	UF	Permeate	100 kDa / PSF / Hollow fiber	
Citric acid		99.7 %	UF	Permeate	100 kDa / PSF / Hollow fiber	
Total Polyphenols	Pomegranate juice	92.0 %	UF	Permeate	150 kDa / Cellulose triacetate / Hollow fiber	Conidi et al., 2017
Cyanidin 3,5-O-diglucoside		90.1 %	UF	Permeate	150 kDa / Cellulose triacetate / Hollow fiber	
Cyanidin 3-O-diglucoside		93.1 %	UF	Permeate	150 kDa / Cellulose triacetate / Hollow fiber	
Delphinidin 3-O-glucoside		82.0 %	UF	Permeate	150 kDa / Cellulose triacetate / Hollow fiber	
Pelargolidin 3,5-O-diglucoside		85.1 %	UF	Permeate	150 kDa / Cellulose triacetate / Hollow fiber	
Anthocyanins		89.7 %	NF	Retentate	2 kDa / Thin film composite / Flat sheet	
Polyphenols		86.0%	NF	Retentate	2 kDa / Thin film composite / Flat sheet	
Sucrose	Pineapple juice	74.2 %	UF	Permeate	100 kDa / PSF / Flat sheet	de Carvalho et al., 2008
Vitamin C	Passion fruit juice	~84.3 %	MF	Permeate	0.3 µm / Polyamide / Hollow fiber	de Oliveira et al., 2012
Galacturonic acid		~59.5 %	MF	Permeate	0.3 µm / Polyamide/ Hollow fiber	
Chlorogenic acid, Cynarin, Apigenin-7-O-glucoside	Artichoke extract	>85.0 %	NF	Retentate	400 Da / PES / Spiral wound	Cassano et al., 2015a
Sinapic acid	Blood orange juice	94.2 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	Destani et al., 2013
p-Coumaric acid		89.9 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Naringin		95.9 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Hydroxybenzoic acid		95.0 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Hesperidin		96.0 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Ferulic acid		93.1 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Epicatechin		95.6 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Ellagic acid		94.7 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Catechin hydrate		97. 0 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Caffeic acid		94.9 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Chlorogenic acid		99.6 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Total phenolics	Castanea sativa leaves aqueous extract	92.1 %	UF	Retentate	5 kDa / modified PES/ Flat sheet	Díaz-Reinoso et al., 2011
		82.5 %	UF	Retentate	10 kDa / modified PES/ Flat sheet	

Soluble protein			UF	Retentate	5 kDa / modified PES/ Flat sheet	
		96.5 %	UF	Retentate	5 kDa / modified PES/ Flat sheet	
		80.5 %	UF	Retentate	10 kDa / modified PES/ Flat sheet	
Total phenols	Apple juice	45.8 %	MF	Permeate	0.45 µm / polyamide /Flat sheet	Fuenmayor et al., 2014
Malic acid		94.8 %	MF	Permeate	0.45 µm / polyamide/ Flat sheet	
Fructose		76.8 %	MF	Permeate	0.45 µm / polyamide/ Flat sheet	
Glucose		85.1 %	MF	Permeate	0.45 µm / polyamide/ Flat sheet	
Sucrose		76.0 %	MF	Permeate	0.45 µm / polyamide /Flat sheet	
Ascorbic acid	Blood orange juice	90.7 %	UF	Permeate	15 kDa / PVDF/ Tubular	Galaverna et al., 2008
Cyanidin-3-glucoside		97.7 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Cyanidin-3-glucoside-6''-malonyl		97.1 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Total anthocyanins		97.6 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Sinapic acid		~100.0 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Caffeic acid		~100.0 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Ferulic acid		~100.0 %	UF	Permeate	15 kDa / PVDF/ Tubular	
p-Coumaric acid		~100.0 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Narirutin		~100.0 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Hesperidin		~100.0 %	UF	Permeate	15 kDa / PVDF/ Tubular	
Total polyphenols	Pineapple juice	92.8 %	MF	Permeate	0.1 µm / PSF/ Hollow fiber	Laorko et al., 2010
		99.6 %	MF	Permeate	0.2 µm / PSF/ Hollow fiber	
		75.0 %	UF	Permeate	30 kDa / PSF/ Hollow fiber	
		91.9 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Polyphenols	Propolis extract	92.8 %	NF	Retentate	98 % rejection Mg SO_4/ Polyamide-polysulphone/ Spiral module	Mello et al., 2010
Flavonoids		~100.0 %	NF	Retentate	98 % rejection Mg SO_4/ Polyamide-polysulphone/ Spiral module	
Polyphenols	Blood orange juice	76.6 %	UF	Permeate	50 kDa / PSF/ Hollow fiber	Mondal et al., 2016
		83.2 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
		76.4 %	UF	Permeate	50 kDa / polyacrylonitrile Hollow fiber	
Anthocyanins		90.7 %	UF	Permeate	50 kDa / PSF/ Hollow fiber	
		92.6 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
		94.2 %	UF	Permeate	50 kDa / polyacrylonitrile/ Hollow fiber	
Histidine	Cactus pear	100.0 %	MF	Permeate	0.2 µm / ceramic/ N.R.	Mosshammer et al., 2006

Glutamine		100.0 %	MF	Permeate	0.2 µm / ceramic/ N.R.	
Isoproline		100.0 %	MF	Permeate	0.2 µm / ceramic/ N.R.	
Proline		100.0 %	MF	Permeate	0.2 µm / ceramic/ N.R.	
Betanin		100.0 %	MF	Permeate	0.2 µm / ceramic/ N.R.	
Isobetanin		100.0 %	MF	Permeate	0.2 µm / ceramic/ N.R.	
Gallic acid	Mate aqueous extract	75.8 %	NF	Retentate	150-300 Da / Thin film/ Spiral-wound	Negrão Murakami et al., 2011
3,4-Dihydroxybenzoic acid		94.7 %	NF	Retentate	150-300 Da / Thin film/ Spiral-wound	
Chlorogenic acid		89.5 %	NF	Retentate	150-300 Da / Thin film/ Spiral-wound	
4,5-Dicaffeoylquinic acid		~100.0 %	NF	Retentate	150-300 Da / Thin film/ Spiral-wound	
Chlorogenic acid	Apple juice	97.7 %	UF	Permeate	100 kDa / PES/ Cassette	Onsekizoglu et al., 2010
Epicatechin		98.5 %	UF	Permeate	100 kDa / PES/ Cassette	
Phloridzin		99.3 %	UF	Permeate	100 kDa / PES/ Cassette	
Citric acid		88.9 %	UF	Permeate	100 kDa / PES/ Cassette	
Galacturonic acid		94.3 %	UF	Permeate	100 kDa / PES/ Cassette	
Malic acid		100.0 %	UF	Permeate	100 kDa / PES/ Cassette	
Quinic acid		96.5 %	UF	Permeate	100 kDa / PES/ Cassette	
Succinic acid		86.2 %	UF	Permeate	100 kDa / PES/ Cassette	
Fumaric acid		90.0 %	UF	Permeate	100 kDa / PES/ Cassette	
Trans-2-hexenal		79.4 %	UF	Permeate	100 kDa / PES/ Cassette	
Gallic acid	Pomegranate juice	87.4 %	UF	Permeate	30 kDa / PVDF/ Cassette	Onsekizoglu et al., 2013
Ellagic acid		84.6 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Catechin		71.1 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Chlorogenic acid		87.7 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Caffeic acid		59.8 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Citric acid		100.0 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Malic acid		100.0 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Quinic acid		92.0 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Oxalic acid		99.6 %	UF	Permeate	30 kDa / PVDF/ Cassette	
Anthocyanins	Blueberry juice	47.6 %	MF	Permeate	0.45 µm /ZrO_2-TiO_2/ Tubular	Pizzolato et al., 2012
Total phenols	Blood orange juice	99.3 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	Quist-Jensen et al., 2016
Naringin		96.2 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Hesperidin		96.8 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Narirutin		95.8 %	UF	Permeate	100 kDa / PSF/ Hollow fiber	
Glycoside	Stevia extract	97.1 %	MF	Permeate	0.05 µm / Al_2O_3-TiO_2/ Tubular	Reis et al., 2009
		93.7 %	MF	Permeate	0.1 µm / Al_2O_3-TiO_2/ Tubular	
		94.7 %	MF	Permeate	0.2 µm / Al_2O_3-TiO_2/ Tubular	
Phenols	Sideritis extract	83.7 %	NF	Retentate	500 Da / modified polyimide/ Flat sheet	Tylkowski et al., 2011
		98.3 %	NF	Retentate	400 Da / polyimide/ Flat sheet	
		99.2 %	NF	Retentate	300 Da / modified polyimide/	

					Flat sheet	
Flavonoids		96.8 %	NF	Retentate	500 Da / modified polyimide/ Flat sheet	
		98.8 %	NF	Retentate	400 Da / polyimide/ Flat sheet	
		99.0 %	NF	Retentate	300 Da / modified polyimide/ Flat sheet	
Anthocyanins	Raspberry	83.0 %	MF	Permeate	0.2 µm / ceramic/ Hollow fiber	Vladisavljević et al., 2013
		56.9 %	UF	Permeate	30 kDa /PSF/ Hollow fiber	
Betalains	Xoconostle fruit juice	> 90.0 %	UF	Permeate	100 kDa /PSF/ Hollow fiber	Castro-Muñoz et al., 2017b
Total polyphenols		> 90.0 %	UF	Permeate	100 kDa /PSF/ Hollow fiber	
Total polyphenols	Pomegranate juice	74.9 %	UF	Permeate	2500 kg mol^{-1}/PSF/ Hollow fiber	Cassano et al., 2015b
		67.3 %	UF	Permeate	2500 kg mol^{-1}/PEEKWC/ Hollow fiber	
Flavonoids	Pomegranate juice	75.8 %	UF	Permeate	2500 kg mol^{-1}/PSF/ Hollow fiber	
		66.5 %	UF	Permeate	2500 kg mol^{-1}/PEEKWC/ Hollow fiber	
Total polyphenols	Pomegranate juice	73.5 %	UF	Permeate	15 kDa/stainless/ Tubular	Baklouti et al., 2012
Total polyphenols	Strawberry juice	97 %	NF	Retentate	150-300 Da/PVDF/ Tubular	Arend et al., 2017
Anthocyanins		99 %	NF	Retentate	150-300 Da/PVDF/ Tubular	
Total polyphenols	Apple juice	48.2 %	UF	Permeate	100 kDa/PSF/ Flat sheet	Domingues et al., 2014
		44.3 %	UF	Permeate	50 kDa/PES/ Flat sheet	Gulec et al., 2017
		63.2 %	UF	Permeate	30 kDa/regenerated cellulose/ Flat sheet	
Total polyphenols	Apple juice	59.2 %	UF	Permeate	100 kDa/modified PSF/ Flat sheet	Gulec, 2018
Cyanindin-3-glycoside	Jussara fruit juice	93.6 %	NF	Retentate	180 Da/polyamide thin film composite/ Flat sheet	Vieira et al., 2018
		97.8 %	NF	Retentate	340 Da/ polyamide thin film composite/Flat sheet	
		89.9 %	NF	Retentate	150-300 Da/ polyamide thin film composite/ Flat sheet	
		78.8 %	NF	Retentate	150-300 Da/polyamide thin film composite/Flat sheet	
		79.9 %	NF	Retentate	400 Da/PES/ Flat sheet	
		50.9 %	NF	Retentate	1000 Da/PES/ Flat sheet	

N.R.: Not reported

Table 4. Biologically active compounds recovered from agro-food by-products using membrane-based technologies.

Recovered compound	*Recovery rate*	*Agro-food by-product*	*Membrane process*	*MWCO/Material/ Configuration*	*Reference*
Phenolic compounds	58.3 %	Orange press liquor	UF	100 kDa / Polysulphone / Hollow fiber	Ruby Figueroa et al., 2011; Ruby Figueroa et al., 2012
	45.7 %	Nixtamalization wastewaters	Integrated membrane process:		Castro-Muñoz and Yañez-Fernandez, 2015; Castro-Muñoz et al., 2016b
			MF	0.2 μm / Polysulfone / Hollow fiber	
			UF	100 kDa / Polysulfone / Hollow fiber	
			UF	1 kDa / Polysulfone / Hollow fiber	
	95.0 %	Olive mill wastewaters	NF	200 Da / Polymeric / Spiral wound	Paraskeva et al., 2007
	11.4 %	Grape seeds	UF	0.22 μm / Cellulose acetate / Flat sheet	Nawaz et al., 2006
	> 70 %	Fermented grape pomace	UF	1000 Da / Thin-film / Spiral wound	Díaz-Reinoso et al., 2009; Díaz-Reinoso et al., 2010
	> 80 %		UF	1000 Da / Ceramic (titania) / Tubular	
	> 30 %		NF	250 Da / Polyamide-polysulfone / Spiral wound	
	> 60 %		NF	350 Da / Polyamide-polysulfone / Spiral wound	
	> 80 %		NF	150-300 Da / Thin-film / Spiral wound	
Hydroxytyrosol, protocatechuic acid, caffeic acid, tyrosol and p-cumaric acid	--	Olive mill wastewaters	MF	0.2 μm / Polypropylene / Tubular	Cassano et al., 2011
	48.3 %		UF	4 kDa / polyethersulphone / Flat sheet	
	21.3 %		UF	5 kDa / Regenerated cellulose / Flat sheet	
	8.7 %		UF	10 kDa / Regenerated cellulose / Flat sheet	
	33.5 %		UF	10 kDa / Polyethersulphone / Flat sheet	
Hydroxycinnamic acids, o-diphenols	81 %	Winery sludge from red grapes	UF	100 kDa / Polysulfone / Flat sheet	Galanakis et al., 2013
	77 %		UF	20 kDa / Polysulfone / Flat sheet	
	56 %		UF	1 kDa / Composite fluoropolymer / Flat sheet	
3,4-DHPEA, p-HPEA, 3,4-DHPEA-EDA, verbascoside, and total phenols	--	Olive mill wastewater	Integrated membrane process:		Servili et al., 2011

			MF	0.3 μm / Polypropylene / Tubular	
			UF	7 kDa / Polyamide-polysulfone / Spiral wound	
p-cumaric	13.3 %	Olive mill wastewaters	Integrated membrane process:		Conidi et al., 2014b
			MF	0.2 μm / Polyvinylidenefluoride / Flat sheet	
			UF	30 kDa / Polysulphone / Hollow fiber	
Chlorogenic acid, Cynarin, Apigenin-7-O-glucoside	100 %	Artichoke wastewaters	Integrated membrane process:		Conidi et al., 2014a
			UF	50 kDa / Polysulfone / Hollow fiber	
			NF	400 Da / Polyethersulfone / Spiral wound	
			NF	150-300 Da / Polyamide / Spiral wound	
	> 85 %	Artichoke wastewaters	NF	400 Da / Polyethersulphone / Spiral wound	Cassano et al., 2015a
Gallic acid, chlorogenic acid and epigallocatechin gallate	100 %	Residues from mate tree	NF	150-300 Da / Thin-film/ Spiral wound	Prudêncio et al., 2012
Free low MW polyphenols, hydroxytyrosol, procatechuic acid, tyrosol, oleuropein, tyrosol and caffeic acid,	> 45 %	Olive mill wastewaters	UF	1 kDa / Polyethersulphone / Spiral wound	Russo, 2007
Proanthocyanidins	> 20 %	Defatted milled grape seeds	UF	200 kDa / Polyvinylidenefluoride / Tubular	Santamaría et al., 2002
Hydroxytyrosol, protocatechuic acid, catechol, tyrosol, caffeic acid, and p-cumaric acid	100 %	Olive mill wastewaters	Integrated membrane process:		Cassano et al., 2013
			UF	0.02 μm / Polyvinylidenefluoride / Hollow fiber	
			UF	1 kDa / Composite fluoropolymer / Flat sheet	
			NF	Salt rejection >97% / Thin-film / Spiral wound	
Isoflavones (aglycone and glucoside)	> 30 %	Soy processing waste	UF	1 kDa / Regenerated cellulose / Spiral wound	Xu et al., 2004
Hydroxytyrosol, procatechin acid, tyrosol, caffeic acid, p-cumaric acid, oleuropein and some other low MW polyphenols.	78 %	Olive mill wastewaters	Integrated membrane process:		Garcia-Castello et al., 2010
			UF	200 nm / Al_2O_3 / Tubular	
			NF	578 Da / Polyethersulphone / Spiral wound	
Hydroxycinnamic acids and flavonols.	13 %	Olive mill wastewaters	UF	100 kDa/ Polysulfone/ Spiral wound	Galanakis et al., 2010

	40 %		UF	25 kDa / Polysulfone / Spiral wound	
	71 %		UF	10 kDa / Polyethersulfone / Spiral wound	
	81 %		UF	2 kDa / Polyethersulfone / Spiral wound	
	99 %		NF	120 Da / Polypiperazine/ Spiral wound	
Anthocyanins, flavonoids	> 90 %	Orange press liquor	NF	180 Da / Polyamide-polysulfone / Spiral wound	Conidi et al., 2012
	> 80 %		NF	300 Da / Polypiperazine amide thin-film composite / Spiral wound	
	> 80 %		NF	400 Da / Polyethersulfone / Spiral wound	
	> 70 %		NF	1000 Da / Polyethersulfone / Spiral wound	
Anthocyanins (cyanidin-3-glucoside chloride, myrtillin chloride and peonidin-3-glucoside chloride), flavanones	> 65 %	Orange press liquor	NF	Na_2SO_4 rejection > 25-50 % / Polyethersulfone / Spiral wound	Cassano et al., 2014
Chlorogenic acid, Apigenin-7-O-glucoside	100 %	Artichoke wastewaters	NF	200-300 Da / Polyamide / Spiral wound	Conidi et al., 2015
Caffeoylquinic acid, flavonoids, chlorogenic acid, cynarin	> 40 %	Artichoke brines	NF	1000 Da / Polyethersulfone / Spiral wound	Cassano et al., 2016a
	> 62 %		NF	400 Da / Polyethersulfone / Spiral wound	
	> 99 %		NF	300 Da / Piperazineamide / Spiral wound	
	> 95 %		NF	150-300 Da / Cross-linked polyamide/ Spiral wound	
	> 93 %		NF	150-300 Da / Cross-linked polyamide/ Spiral wound	
Phenolic compounds	> 80 %	White vinasses	NF	200 Da / Polyethersulfone / Spiral wound	Díaz-Reinoso et al., 2017
Phenolic compounds	21 %	Winery effluents	MF	0.4 μm / Polyimide / Hollow fiber	Giacobbo et al., 2015
	5 %	Winery effluents	MF	0.2 µm / PVDF / Hollow fiber	Giacobbo et al., 2017b
Phenolic compounds	>90 %	Racking wine lees	Integrated membrane process: UF	 10 kDa/Fluoropolymer/Tubular	Giacobbo et al., 2017a

			UF	1000 Da/ Fluoropolymer /Tubular	
			NF	200-300 Da/Polypiperazine/Tubular	
	57 %	Olive mill wastewaters	UF	3 kDa/Regenerated cellulose/Flat sheet	Ochando-Pulido and Martínez-Férez, 2017
Phenolic compounds	97-98 %	Apple pomace extract	NF	150-300 Da/Polyamide thin film composite/Spiral wound	Uyttebroek et al., 2018
Quinic acid	92 %	Apple pomace extract	NF	150-300 Da/Polyamide thin film composite/Spiral wound	
Catechin	78 %	Apple pomace extract	NF	150-300 Da/Polyamide thin film composite/Spiral wound	
Epicatechin	87 %	Apple pomace extract	NF	150-300 Da/Polyamide thin film composite/Spiral wound	

MW: Molecular weight

Table 5. Permeate water streams obtained from the fractionation of agro-food by-products by integrated membrane processes (adapted from Castro-Muñoz et al., 2017a).

Agro-food by-product:	Integrated membrane process conformed by: (Membrane operation: MWCO)	Permeate sample obtained:	Characteristics of permeates:	Reference:
Olive mill wastewater	UF: 0.2 μm UF: 1000 Da NF: >97 % $MgSO_4$ rejection		Low TOC: 95 mg L^{-1} Low TC: 100 mg L^{-1} Low TIC: 5 mg L^{-1} Hydroxytyrosol: 0 mg L^{-1} (N.D.) Caffeic acid: 0 mg L^{-1} (N.D.) p-cumaric acid: 0 mg L^{-1} (N.D.) Tyrosol: 0 mg L^{-1} (N.D.) Catechol: 0 mg L^{-1} (N.D.) Protocatechuic acid: 0 mg L^{-1} (N.D.) Total phenols: 0 mg L^{-1} (N.D.)	Cassano et al., 2013
Orange press liquor	UF: 100 kDa NF: 25-50 % Na_2SO_4 rejection	Not reported	Low TSS: 4.5 g 100 g^{-1}	Conidi et al., 2012
Artichoke wastewaters	UF: 50 kDa NF: 400 Da, 85-95 % Na_2SO_4 rejection NF: 150-300 Da, 96 % $MgSO_4$ rejection	Not reported	Glucose: 0 mg L^{-1} (N.D.) Fructose: 0 mg L^{-1} (N.D.) Sucrose: 0 mg L^{-1} (N.D.) Cynarin: 0 mg L^{-1} (N.D.) Chlorogenic acid: 0 mg L^{-1} (N.D.) Apigenin-7-O-glucoside: 0 mg L^{-1} (N.D.)	Conidi et al., 2014a
Artichoke wastewaters	UF: 100 kDa NF: 400 Da, 85-95 % Na_2SO_4 rejection NF: 150-300 Da	Not reported	Glucose: 0 mg L^{-1} (N.D.) Fructose: 0 mg L^{-1} (N.D.) Sucrose: 0 mg L^{-1} (N.D.) Cynarin: 0 mg L^{-1} (N.D.) Chlorogenic acid: 0 mg L^{-1} (N.D.) Apigenin-7-O-glucoside: 0 mg L^{-1} (N.D.)	Cassano et al., 2015a

Nixtamalization wastewaters	MF: 0.2 μm UF: 100 kDa UF: 1000 Da		Low TOC: 381 mg L^{-1} Low Carbohydrates content: 0.26 mg mL^{-1} Low Turbidity: 3.78 NTU Free TSS: 0 ºBrix (N.D.)	Castro-Muñoz and Yañez-Fernandez, 2015; Castro-Muñoz et al., 2016

TOC: Total Organic Carbon; TC: Total Carbon; TSS: Total Soluble Solids; TIC: Total Inorganic Carbon; N.D.: Not detected.

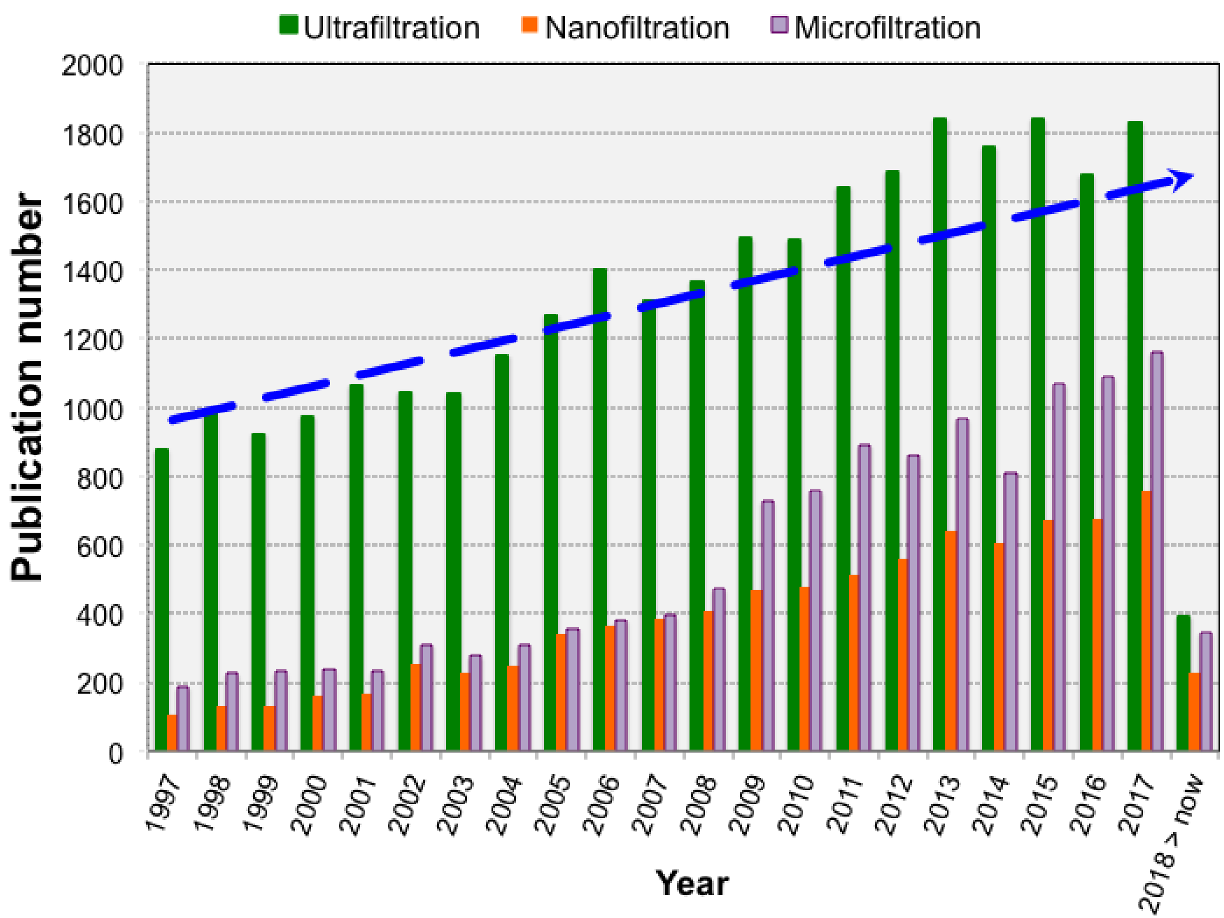


Figure 1. Evolution of the use of membrane-based technologies for the processing of food and by-products (by Scopus, April 28th, 2018).

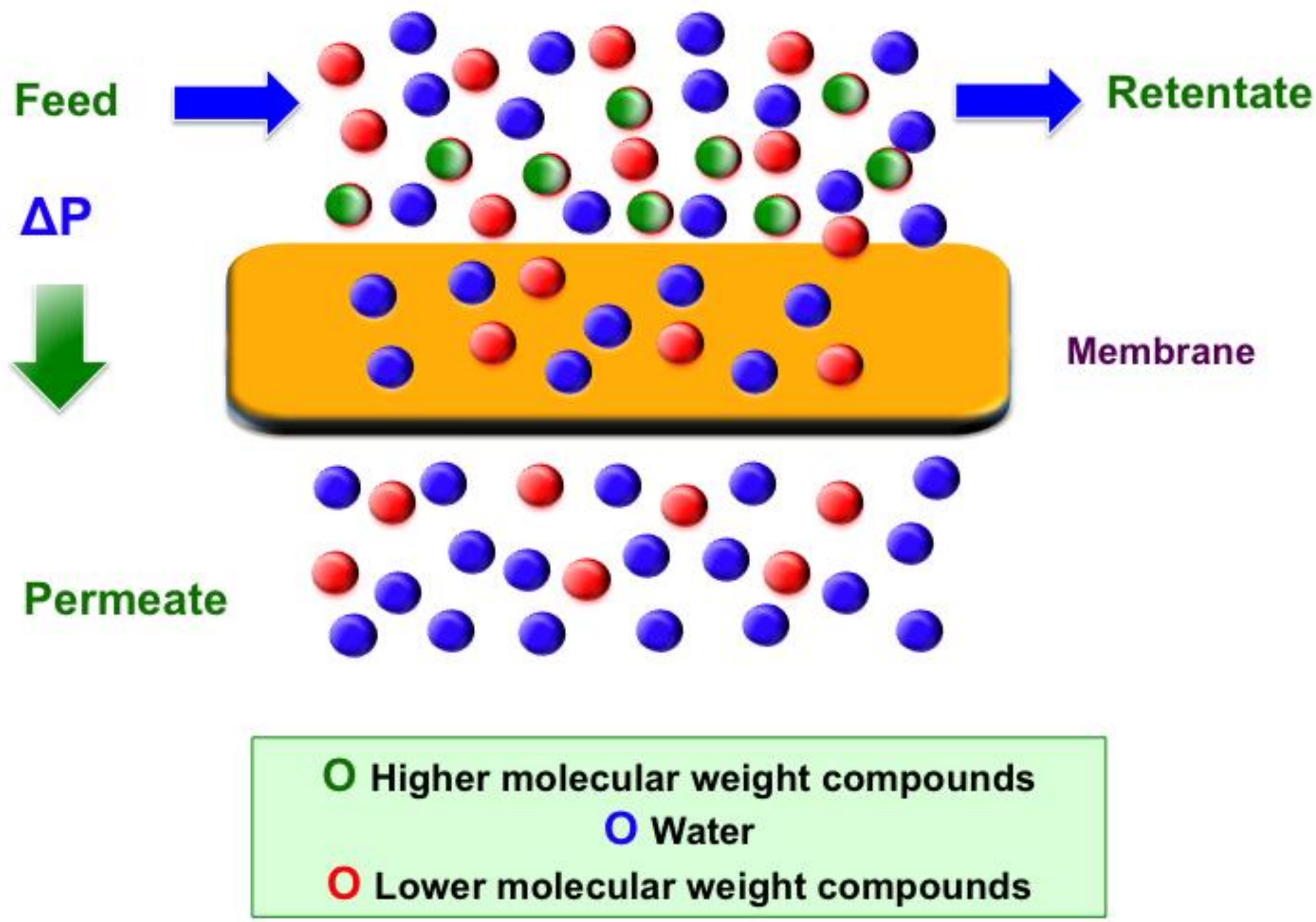


Figure 2. General drawing of pressure-driven membrane technology.

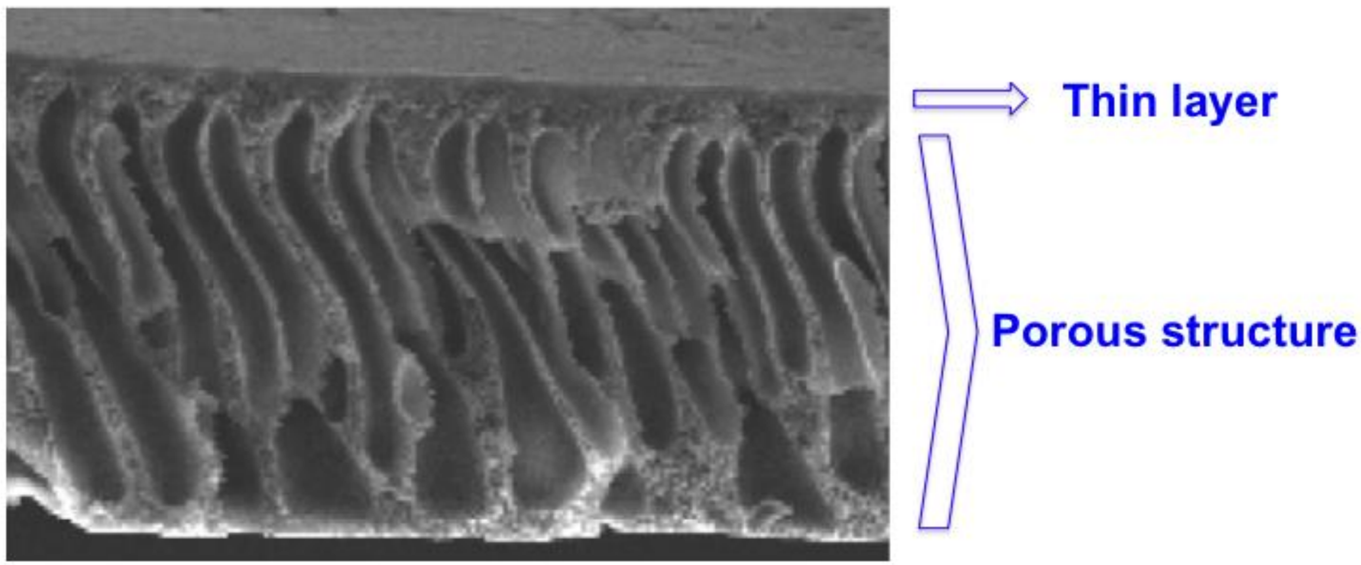
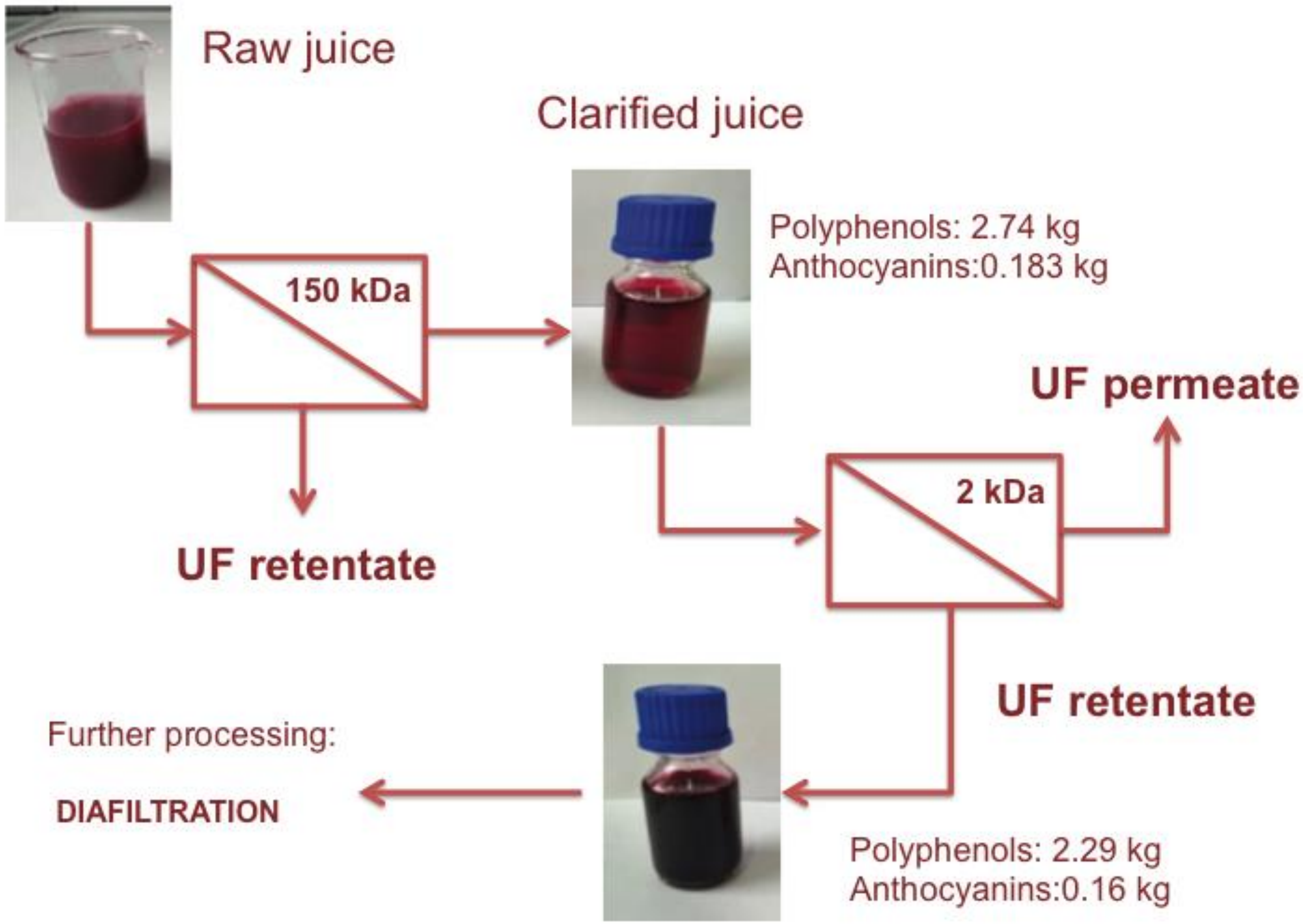


Figure 3. Cross section view of an asymmetric UF membrane (Ali et al., 2011).



Raw juice
Clarified juice
150 kDa
Polyphenols: 2.74 kg
Anthocyanins:0.183 kg
UF permeate
2 kDa
UF retentate
UF retentate
Further processing:
DIAFILTRATION
Polyphenols: 2.29 kg
Anthocyanins:0.16 kg

Figure 4. Integrated membrane process proposed for the recovery of phenolic compounds and anthocyanins from pomegranate juice (adapted from Conidi et al., 2017).

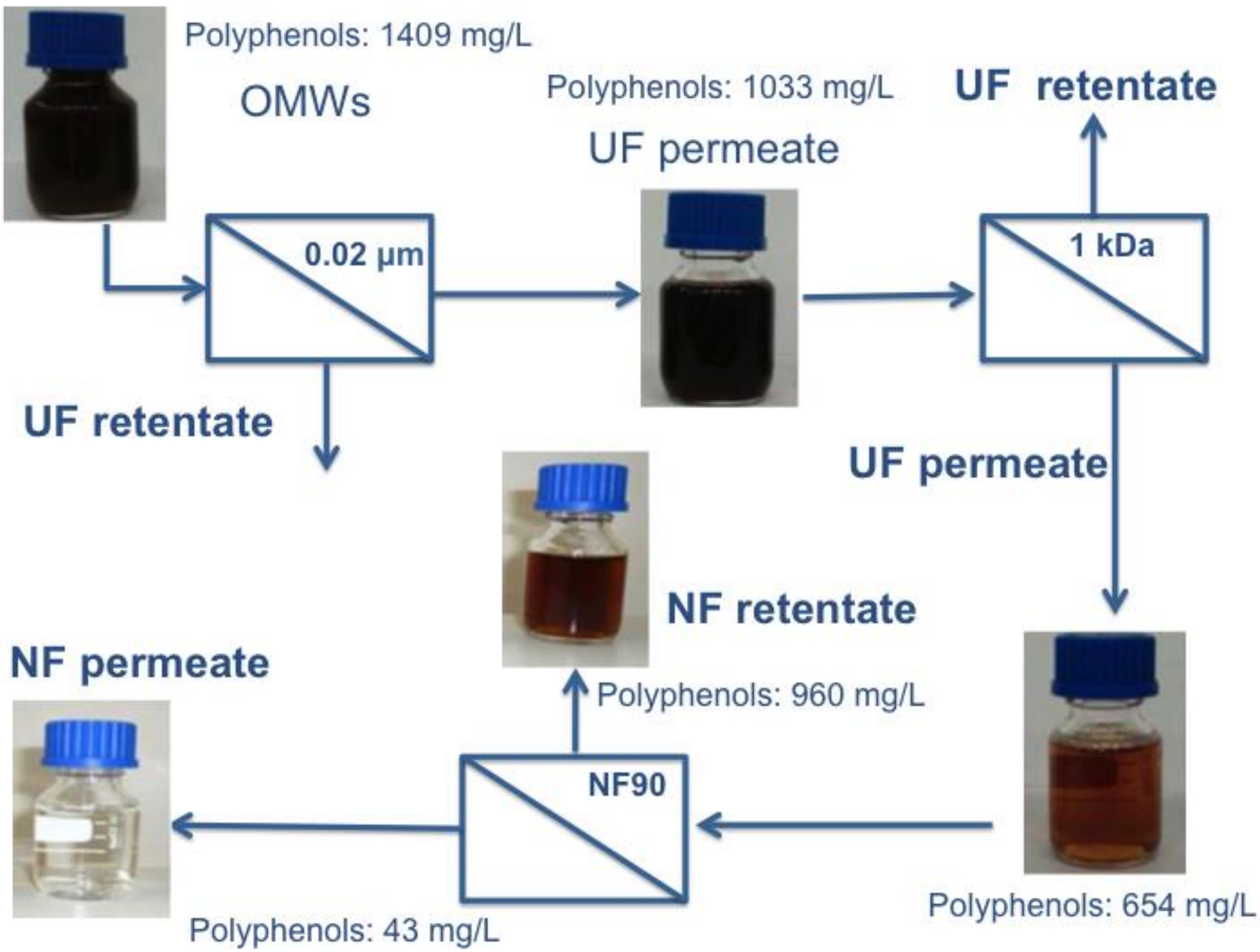


Figure 5. Integrated membrane process proposed for the recovery of phenolic compounds from OMW; NF90 membrane >97% $MgSO_4$ salt rejection (adapted from Cassano et al., 2013).

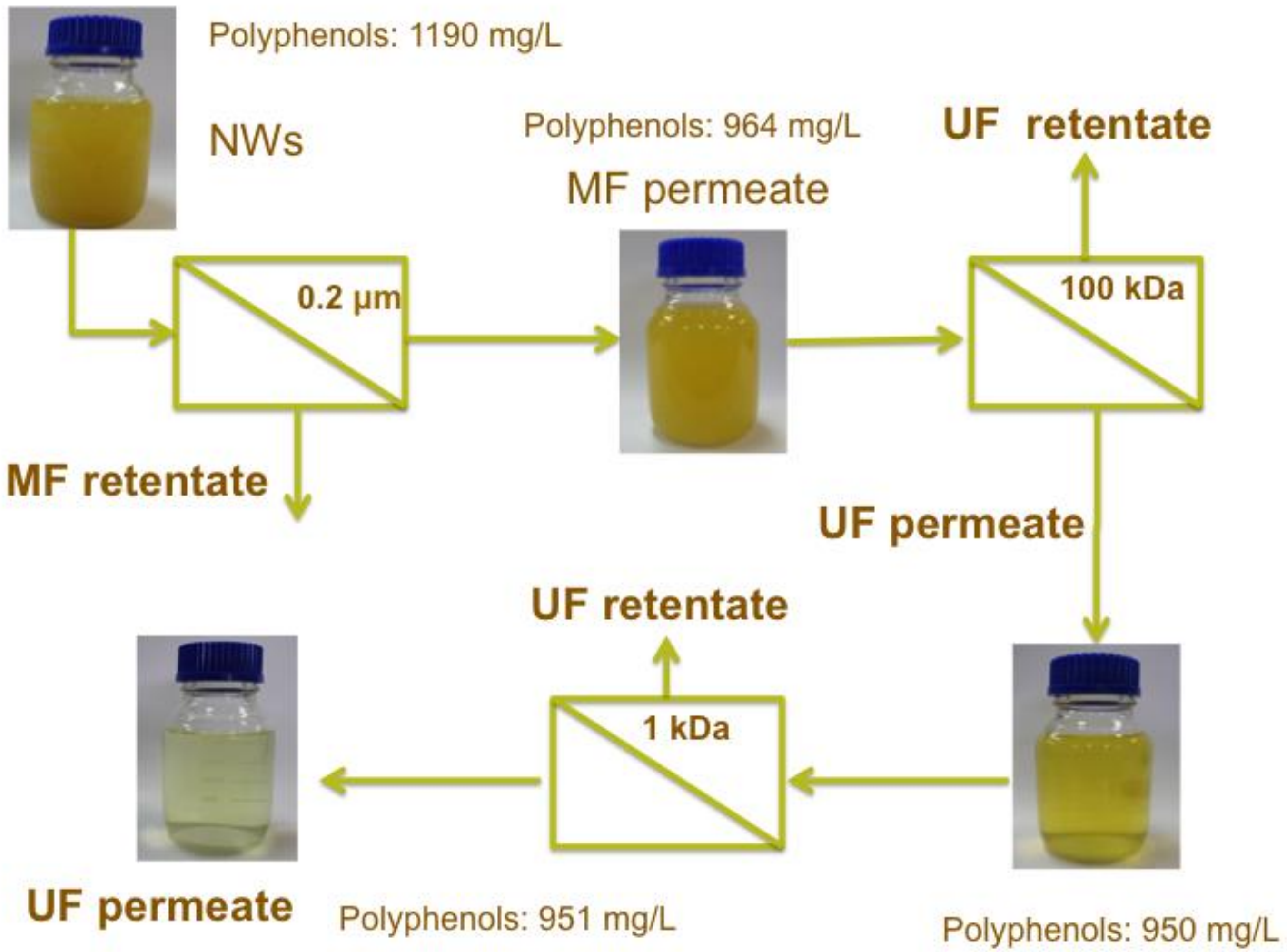


Figure 6. Integrated membrane process proposed for the recovery of phenolic compounds from NWs (adapted from Castro-Muñoz and Yañez-Fernandez, 2015)**.**

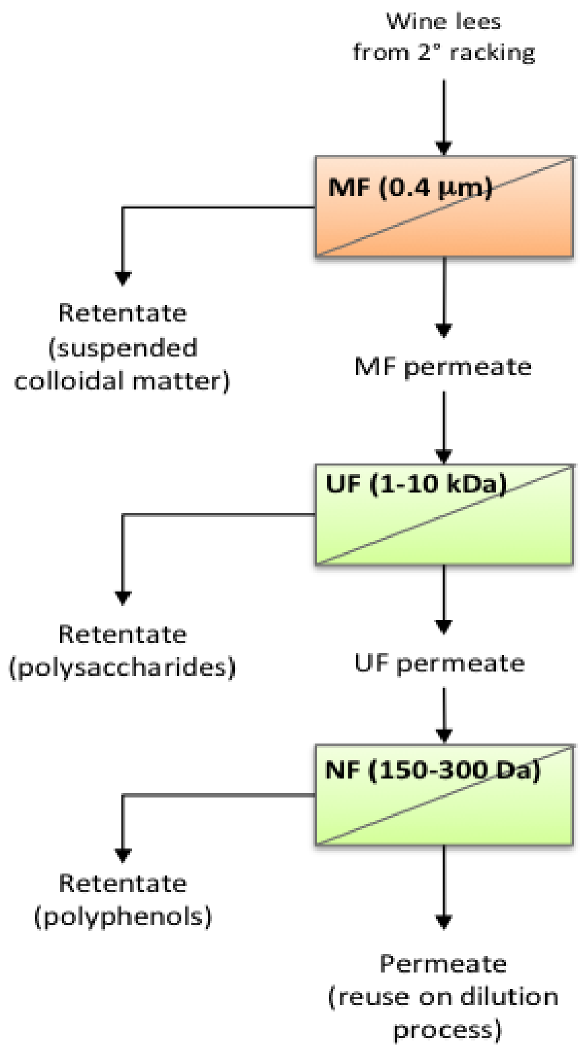


Figure 7. Integrated membrane process for the fractionation of wine lees (adapted from Giacobbo et al., 2017a).