Chapter 11
Coupling of Membrane Technology with Emerging Technologies for the Recovery of Bioactives

D. Shanthana Lakshmi, K. S. Radha, and S. Ananda Kumar

Abstract In this chapter an overview of the bioactive extraction techniques by the combination of membrane processes and coupling it with new emerging technologies is provided. The main classes of bio actives, their classification with their potential health benefits is discussed. The importance of food bio actives, increasing consumer awareness and their economical extraction processes without significant loss in their original characteristics are highlighted. Specific applications of different membrane unit operations and their multi stage integration in certain selected areas of bioactive extraction of natural sources (artichoke waters, olive oil mill waters, blood orange Juice, Pomegranate juice, whey waters) are also reviewed and discussed, The potential of membrane techniques with respect to the separation, concentration and retention of high-added-value compounds such as Phenolics flavonoids, polyphenols, lactoferrin and their vital role in food quality improvement and reduction of environmental foot print are analyzed in detail.

Keywords Bioactives · Integrated membrane process · Ultrafiltration · Microfiltration · Nanofiltration · Osmotic distillation · Vacuum membrane distillation · Reverse osmosis · Microwave extraction · Pressurized liquid extraction · Encapsulation · Polyphenols · Flavonoids · Lactoferrin

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1 Introduction

Over recent years, consumers have become more concerned about the quality of ingredients being used in foods and beverages (Galiano et al. 2019; Castro-Muñoz 2019). The growing prevalence of risk factors, such as stress, obesity, diabetes, and high blood pressure, has increased the awareness about the need for the adoption of a healthy diet to stay healthy and fit. Apparently, plant-based bio-actives play a vital role, which are vegan extracts having high potential usage in food and beverages, dietary supplements, animal nutrition, and personal care products as well (Castro-Muñoz et al. 2020a, b; Castro-Muñoz 2020a). The rising trends in naturally sourced products along with a preference for plant-based derivatives over animal-sourced will positively influence the industry's expansion.

In addition, a systematic nutritional approach, including the administration of food bio-actives and micro-nutrients, also have the potential to augment immune function and defend against the prevailing COVID-19 pandemic. The supplementation of well-established antioxidant, antiviral, antimicrobial, anti-inflammatory and cytotoxic properties of food bio-actives such as polyphenols, flavonoids, carotenoids have shown to be exceptionally beneficial in enhancing immunity against several viral infections (Cassano et al. 2018; Valencia-Arredondo et al. 2020; Díaz-Montes et al. 2020a).

Owing to the above said increasing consumer awareness and conscience to use vegetarian products have resulted in an increase in plant-based bio-actives market growth. These factors are encouraging vendors to introduce innovative products that have high nutritional value, thereby fuelling the market growth. According to the latest market research report published by Global Market Estimates (GME), a high Compound Annual Growth Rate (CAGR) of 8.27% for the plant-based bio-actives over the forecast period from 2021 to 2026.

1.1 Bioactive Extraction

Food waste is the potential bioresource for the extraction of nutraceuticals and bioactive compounds. Various investigations have demonstrated that the food wastes obtained from sources like fruits, vegetables, cereal and other food processing industries can be used as potential sources of bioactive which has significant application in treating various ailments (Castro-Muñoz et al. 2018; Tarazona et al. 2018; Cassano et al. 2016). Food waste is generated in all stages of the food life cycle (Castro-Muñoz et al. 2017a). Up to 42% of food waste is produced by household activities, 39% losses occurring in the food manufacturing industry and 14% in foodservice sectors such as restaurants, catering services, tiffin centers, etc. while 5% is lost during distribution. Food waste is expected to rise to about 126 Mt. by 2020 if any prevention policy or activities are not undertaken (Mirabella et al. 2014). Significant action is being taken to achieve the prevention of multiplying wastes through the extraction of high-value components from them which can be re-used as nutraceuticals and functional ingredients. Bioactive components present in such agricultural waste can be recovered using various techniques.

1.2 Advantages of Membrane Technology

Membrane technologies are the most promising tool that appears as a valid approach to bioactive extraction from natural resources having been identified for their varied benefits compared to the existing conventional techniques (Castro-Muñoz 2020b; Díaz-Montes et al. 2020b). The major key points which include less time and energy consumption, zero use of chemical or biological additives, modular and simple design, optimum pressure, temperature conditions, without loss of biological properties in the extracts and very minimal risk of product contamination (Díaz-Montes et al. 2020a; Castro-Muñoz et al. 2019a; Haq et al. 2021). They are also characterized by very high selectivity and specificity with respect to separation involving very easy steps of processing (Castro-Muñoz et al. 2018). Especially, the pressure-driven techniques such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) are the emerging technologies for clarification, fractionation, concentration and separation of bio-actives from food products and their derivatives due to their intrinsic properties and sustainable attributes. Further to this approach, other state of art technologies, such as pervaporation (PV) (Galiano et al. 2019; Castro-Muñoz 2020b; Castro-Muñoz et al. 2020c), osmotic distillation (OD) (Conidi et al. 2020), membrane distillation (MD) (Gontarek et al. 2019, 2021), are also employed in recent years for the recovery strategies (Conidi et al. 2020).

1.3 Recovery of Bioactives Based on Membrane Technologies

Membrane technology has proven to be an ideal alternative to the traditional juice clarification and UF and MF treatments are successfully used for processing fruit and vegetable juices (Castro-Muñoz et al. 2016; Galanakis et al. 2016). Ideally, UF clarifies and concentrates the bioactives based on the molecular weight cut off of the membranes and it also clears and segregates the components i.e., microorganisms, colloids, proteins, tannins, yeast, moulds etc. that pollutes and thereby preserves biologically properties of the final stable extract. While MF facilitates mainly juice clarification (Cassano et al. 2019). Results reveal that there is more membrane fouling in UF conditions than in MF operations (Pichardo-Romero et al. 2020). Hence, MF has been used as a pre-treatment step in the extraction process in order to reduce the fouling encountered in UF membranes. Both the processes operate at different efficiency levels in the recovery process. Nanofiltration (NF) membranes are utilized for fruit juice concentration to regulate sugar concentration and also to separate phenolic compounds from sugars (Castro-Muñoz et al. 2019a; Cassano et al.

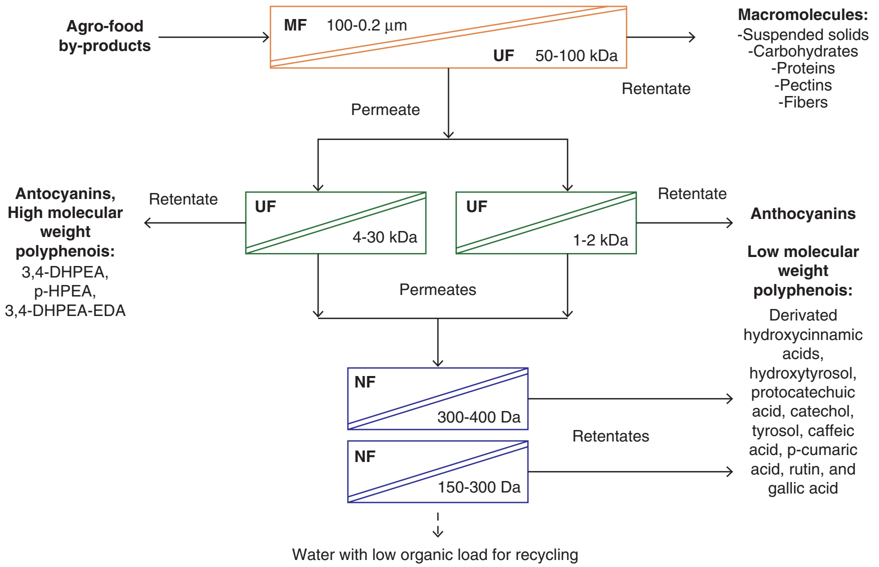

Fig. 11.1 Recovery of high-added value components by integrated membrane processes (Castro-Muñoz et al. 2019a)

2015). As a general overview, Fig. 11.1 illustrates the various bioactive compounds recovered and the Table 11.1 enlists the recovery rate of such pressure-driven membrane technologies towards various bioactive compounds.

Bioactives generated have confirmed the essence of fruits' complete health benefits due to the presence of phenolic attributes containing remarkably a higher dosage of hydrolyzable tannins, as well as anthocyanins exhibiting high antioxidant activity (Castro-Muñoz 2019). Hence, membrane processes are considered the leading technology and very efficient unit operations in the recovery of food bioactives, such a phenolic compound, antocyanins, peptides, amino acids, among others (Castro-Muñoz et al. 2021a). In the recent past, a novel and innovative approach arise from the combination of

(i) Different membrane unit operations (UF, MF, NF, RO, OD, MD) (Gontarek-Castro et al. 2021; Castro-Muñoz et al. 2019b, 2021b) or
(ii) Membrane operations and conventional separation technologies (Soxhlet, maceration, hydro distillation) or
(iii) Membrane-based technologies with emerging technologies, such as ultrasound, microwave with a rationale of intensification of the extraction process (Drioli and Romano 2001).

As shown in Figs. 11.2 and 11.3 the main class of bioactive compounds including polyphenols, carotenoids, flavonoids, vitamins, omega-3 fatty acids, tannins and organic acids, have attracted great attention due to their role in the prevention of several chronic diseases. Also, Fig. 11.4 reports the main biological activity of such

Table 11.1 Recovery rate of MF, UF and NF membranes towards several biomolecules (Castro-Muñoz et al. 2019a)

No.	Membrane technology	Recovery rate	Bioactives generated
1.	MF	47–100% in permeate	Anthocyanins, glutamine, isoproline, proline, betanin, isobetanin, sugars, galacturonic acid and some phenolic compounds.
2.	UF	44–99% in permeate	Anthocyanins, glutamine, isoproline, proline, betanin, isobetanin, sugars, galacturonic acid and some phenolic compounds.
3.	NF	50–99% in retentate	Anthocyanins, glutamine, isoproline, proline, betanin, isobetanin, sugars, galacturonic acid and some phenolic compounds.

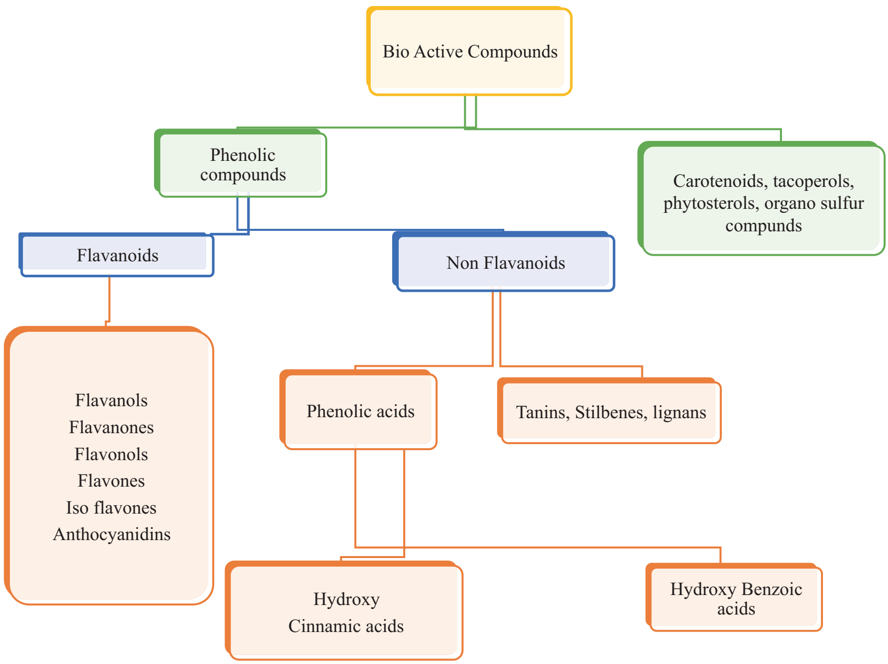

Fig. 11.2 The main classes of bioactive compounds

high-added value compound and their main sources in natural products. They are extra-nutritional constituents that are found in small quantities in foods providing health benefits besides the basic nutritional value of the product. The extraction of bioactive compounds starts with the selection of suitable methods, protocols, sample preparation, and extensive literature survey. During the extraction, the major concern lies in minimizing the interference of unwanted materials that may co-extract with the focused compounds. A number of extraction techniques have been introduced along with the existing traditional classical extraction methodology

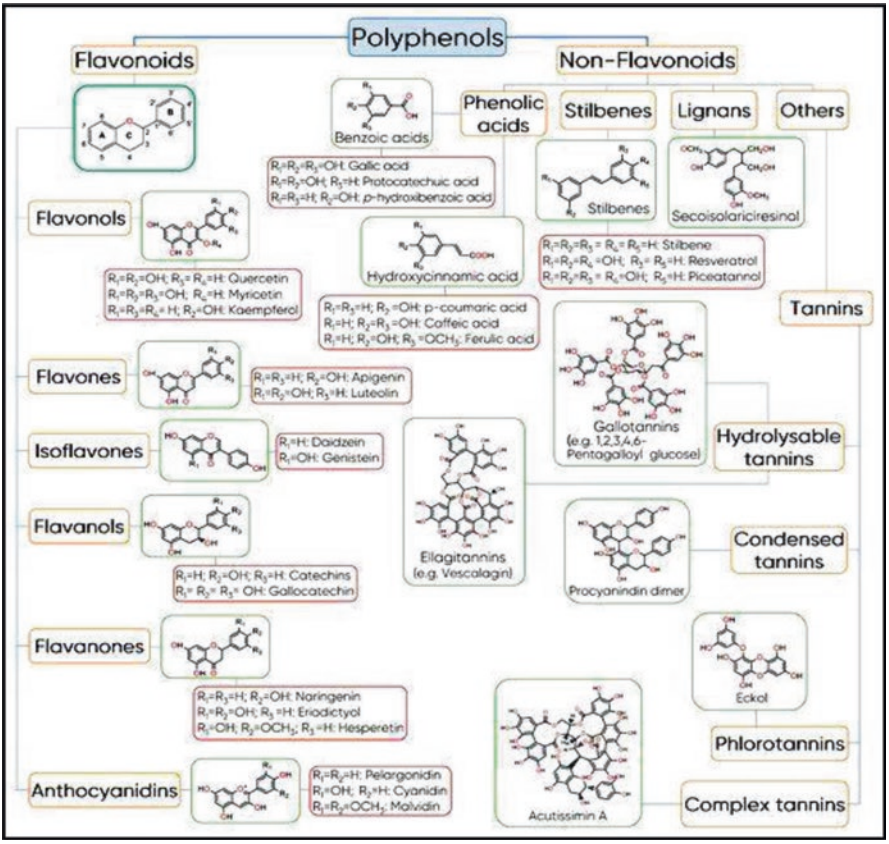

Fig. 11.3 Classification and structure of polyphenols (Câmara 2021)

(Valencia-Arredondo et al. 2020; Díaz-Montes et al. 2020b). But researchers are in the process of developing an eco-friendly single standard energy-saving method for extracting bioactive compounds from food wastes. There are various parameters like the nature of the plant matrix, the chemistry of bioactives, and scientific expertise that influence the efficiencies of conventional and emerging technological extraction.

2 Combined Membrane Unit Operations and Bioactive Food Products

Integrating the membrane technologies for the recovery of bioactives is recommended as the best method in place of traditional technologies. The following are the examples that indicate the importance of integrated membrane operational processes towards bioactive recovery.

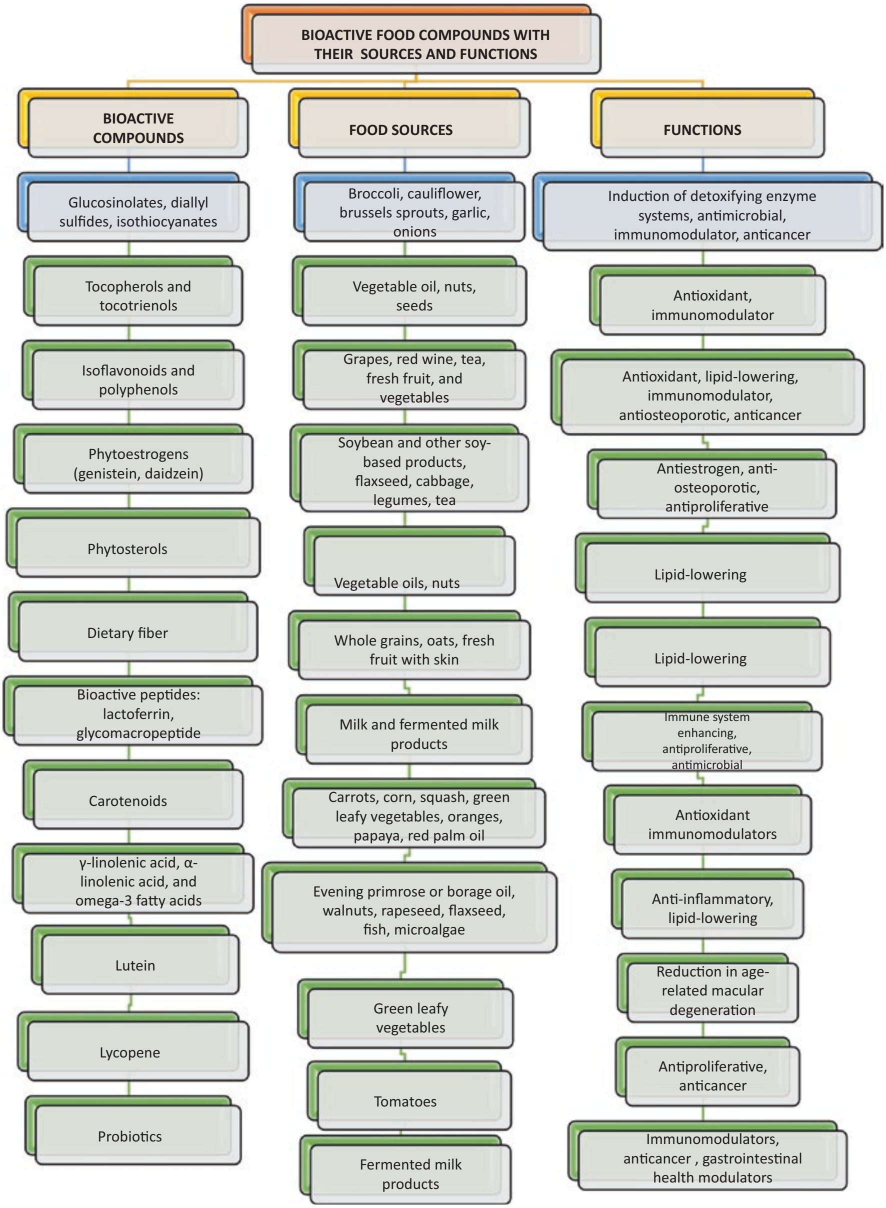

Fig. 11.4 Bioactive food compounds with their sources and functions

2.1 Two Stage Integrated Process

MF and UF processes are coupled to study the recovery of various bioactive compounds in different combinations. Laorko et al. (Laorko et al. 2010) established that the 0.2 μm polysulfone MF membrane was best suited for the treatment of pineapple juice due to the highest permeate flux and the highest recovery of polyphenols and other antioxidant compounds in comparison to UF membranes (MWCO in the range of 30-100 kDa) in a hollow fiber configuration. One more analysis was performed by Cassano et al. (2010) with PVDF MF (0.2 μm) and UF membranes with MWCO of 200 kDa in the flat-sheet configuration on the physicochemical composition of cactus pear juice, indicated better performance of the MF process in terms of permeation flux and recovery of phenolic compounds in the permeate stream. However, both membrane processes permitted a clarified juice with enriched physicochemical and nutritional properties comparable to those of the fresh cactus juice. Similarly, there are several other investigations with UF membranes variation in the membrane material (PS, PVDF) or configuration (flat sheet, spiral wound, hollow fiber) or of MWCO (0.15 kDa to 150 kDa) for the extraction of bioactive. In all

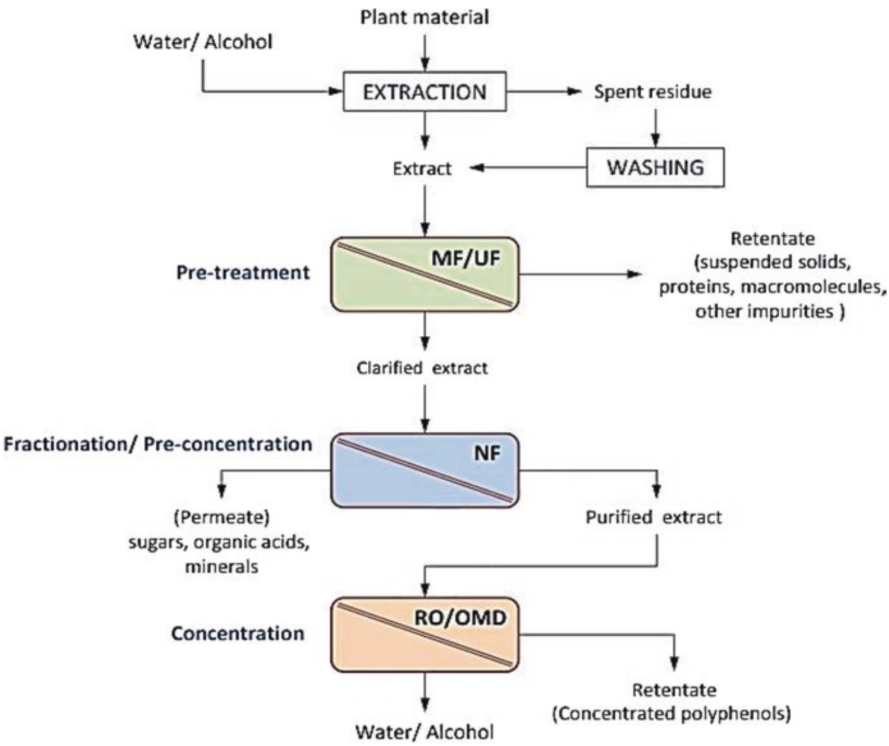

Fig. 11.5 General flow sheet for the recovery of phenolic compounds from vegetable sources by membrane processing (Conidi 2018)

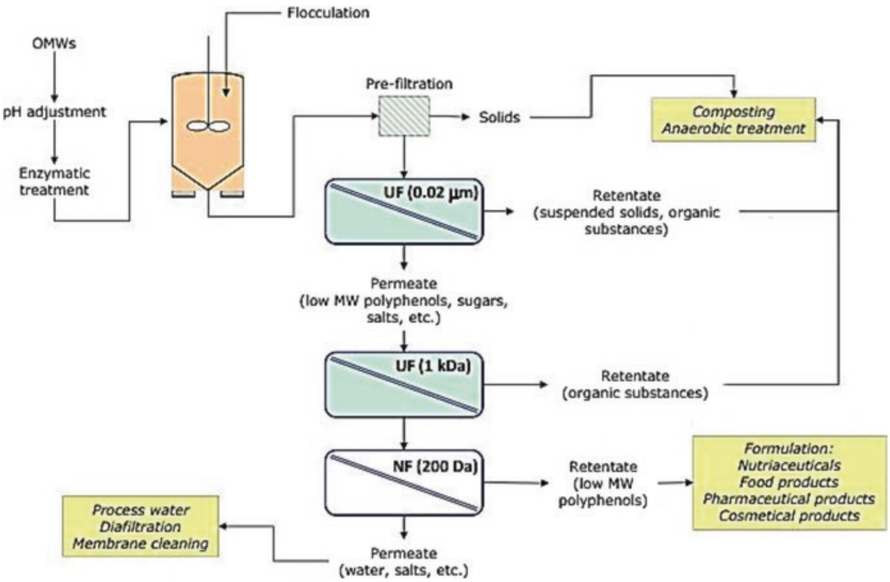

Fig. 11.6 Conceptual proposed design for the recovery of polyphenols from olive mill wastewaters (Conidi 2018)

these investigations, observed retentions of total anthocyanins, total polyphenols and TAA were in the range of 80–97%, Thus the pressure-driven membrane processes are becoming a real alternative to traditional separation systems for recovering the bioactive compounds (Conidi et al. 2018) (Figs. 11.5 and 11.6).

Cassano et al. (2011) presented a novel integration of the UF and OD process for the production of concentrated pomegranate juice enriched with bioactive compounds. Fresh fruit juice clarified through hollow fiber UF membranes then concentrated by OD. The highlights of Cassano's works are summarized in Table 11.2**.**

Cisse et al. (2005) subjected the Orange juice cross-flow microfiltration (CFM) through a 0.2 μm ceramic membrane. The clarified orange juice (permeate) was then concentrated at low temperatures by osmotic evaporation (OE) in two stages. The integrated process, of CFM together with OE, represents an attractive technical and ideal alternative to thermal technologies because it preserves the juice's original quality better and progressively increases the vitamin C content of the concentrate towards the levels found in the initial juice without comprising the original color of the product. In another work, Alves and Coelhoso (2006) made a comparison between the two processes namely OE and Membrane distillation (MD) and concluded from the results that OE is the best alternative for orange juice extraction in terms of aroma retention, mass transfer resistance and water flux.

Balyan and Sarkar (2016) evaluated the potential of an integrated membrane process, by employing a suitable combination of membrane systems to extract phenolic compounds from Jamun seed extract using water as a solvent. In particular, aqueous Jamun seed extract undergoes a preliminary treatment process using UF

Table 11.2 Process integration of different membrane-based technologies for the recovery of bioactives from pomegranate juice (Cassano et al. 2011)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
UF coupled with OD Natural source: Pomegranate juice Two stage process	First step is the clarification of non-depectinized juice by hollow-fiber UF membranes; The second step is the concentration of the clarified juice by using an OD apparatus. Operating conditions: Ambient temperature (25 ± 2 C) Total soluble solids (TSS) content of 162 g kg1 and 520 g kg1, respectively	Organic acids (malic, ascorbic and citric acids), total polyphenols and anthocyanins (cyanidin 3,5-diglucoside, delphinidin 3- glucoside, etc.)	Clarification results in unchanged physico-chemical and nutritional properties as those of the fresh juice by UF The total antioxidant activity of the OD retentate at 52 °brix was only 4% lower than the TAA of the clarified juice. The antioxidant activity of pomegranate juice, attributed largely to total phenols and anthocyanins content preserved more efficiently during the concentration step independently over the level of total soluble solids obtained.

membranes, followed by a concentration using an NF process. Table 11.3 reports the main insights and concluding remarks provided by Balyan and Sarkar (2016).

2.2 *Three Stage Integrated Process*

Torun et al. (2014) recently examined an integrated membrane process including MF, RO, OD for producing concentrated sage (*Salvia fruticosa* Miller) extract for the recovery of polyphenols, as detailed in Table 11.4. Dried sage leaves (*Salvia fruticosa* Miller) are processed using hot water. An integrated membrane process was employed to extract the concentrate (32.4 w/w%). Retention and loss studies were carried out for the determination of a number of total polyphenols, flavonoids and also the antioxidant activity of the recovered bioactives. Finally, the composition of phenols in the final extract was identified with the HPLC technique.

In a similar way, Conidi et al. (2014) analyzed another three-stage membrane integrated process on artichoke wastewaters by using one UF and two different NF membranes in a sequential combination (see Table 11.5). The authors proposed the recovery of bioactives from artichoke derivatives since this natural source contains different biomolecules with potential health benefits, as represented in Fig. 11.3. As illustrated in Fig. 11.4**,** the strategy implied a smart sequence of various types of

Table 11.3 Process integration of different membrane-based technologies for the recovery of bioactives from pomegranate juice (Balyan and Sarkar 2016)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
UF coupled with NF Natural source: Jamun (Syzygium cumini L.) seed extracts Two stage process	First step in the process is cross flow ultrafiltration for initial clarification, followed by concentration using nanofiltration under batch concentration mode. Operating conditions: Optimal condition (temperature: 49.2 °C, time: 89.4 min, and liquid to solid ratio: 51.6:1 mL/g)	Phenolic compounds – Poly phenols and flavonoids	Experimental results revealed that purity of the UF clarified phenolic extract was increased from 39.2 to 53%. Further, the UF clarified extract, with an initial total polyphenol content of 942 mg GAE/L, was concentrated by nanofiltration up to a factor of 3.2 This membrane showed a retention towards total polyphenols and total flavonoids of 78 and 100%, respectively. NF retentate showed higher antioxidant activity (94.6%) of inhibition than the UF permeate showing 75.5 ± 1.8% of inhibition by DPPH method and FRAP method results 530 ± 10 μM Fe(II)/L and 430 ± 10 μM Fe(II)/L of higher polyphenols content.

membranes, along with adsorption processes for the successful recovery of the compounds (Figs. 11.7 and 11.8).

By comparing several works developed by Cassano's group, Fig. 11.9 shows a comparison of the rejection rates towards various phenolics using different integrated processes. In general, chlorogenic acid, cynarin and apigenin-7-O- were completely rejected by most of the proposed recovery techniques (Fig. 11.10).

Integrated membrane processes targeting to remove sugar compounds from phenolic compounds have also been proposed for by-products of the citrus processing industry, such as orange press liquor and bergamot juice. Similar to Artichoke, citrus fruits, such as blood orange, also contains plenty of biomolecules with a related biological activity, as can be seen in Fig. 11.11. In the experiments, an integrated process (Cassano et al. 2014) involving based on the use of UF, NF and OD processes for the recovery and concentration of flavonoids from orange press liquor was investigated and reported accordingly. This UF process allowed removing all suspended solids from the raw press liquor while flavonoids and anthocyanins were recovered in the clarified fraction (rejections towards flavanones and anthocyanins were lower than 1%). The NF process produced concentrated extracts enriched in

Table 11.4 Process integration of different membrane-based technologies for the recovery of bioactives from *Salvia fruticosa* Miller extract (Torun et al. 2014)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
MF coupled with RO and OD Natural source: Sage (Salvia fruticosa Miller) extracts. Three stage process	In first stage, they used a multi-tubular ceramic MF membrane for concentration of preliminary bioactive extract. Operating temperature (30 °C) and fixed recirculation flow rate (500 Lh^{-1}). The permeate from the MF stream was pre-concentrated by using RO flat-sheet membrane. Operating temperature (30 °C) and fixed recirculation flow rate (600 L h^{-1}). And the last procedure is done with osmatic distillation using poly propylene hollow fibre membrane module.	4 phenolic compounds (caffeic-acid, p-coumaric 8 acid, ferulic acid and rosmarinic acid) and 4 flavonoids (rutin, luteolin, hesperetin and 9 apigenin and Flavanoids	It is well observed that all the biochemical properties i.e., total phenolic content, total flavonoid content and anti-oxidant activity possessed around 60% of retention during RO process (63%, 56%, 56%, respectively) and during OD process it was observed to reach higher than that 90% of retention (95, 96, 99%, respectively).

bioactive compounds due to the high rejection measured flavanones (97.4%) and anthocyanins (98.9%) (Figs. 11.12 and 11.13, Table 11.6).

Conidi et al. (2011) investigated the extraction and concentration of phenolic compounds from bergamot juice (a by-product of essential oil production) using UF and NF membranes, as described as follows (Table 11.7):

The fractionation of phenolic compounds from sugars in clarified bergamot juice was also investigated by using three NF membranes (NF PES10, N30F and NF270) in spiral-wound configuration with varying MWCO (150, 400 and 1000 Da) and polymeric material (Polypiperazine amide and PES) (Conidi and Cassano 2015). For NF PES 10 membrane, with an MWCO of 1000 Da, measured rejection of flavonoids (naringin, nehesperidin and hesperidin) was found to be between 88.4 and 90.1%, while the rejection of sugars was 35%.

In an attempt with tight UF and NF membranes to recover specific derivative phenolic compounds, such as hydroxytyrosol, catechol, tyrosol, caffeic and p-cumaric acids, displayed over 80% recovery rate with high antioxidant activity (2175 mg L-1 Trolox). A conceptual process design for the recovery of phenolic compounds from OMWs was proposed by Cassano et al. (2013) (Figs. 11.14, 11.15, 11.16, and 11.17, Table 11.8).

In this context of integrated membrane techniques, a combination of NF and RO membranes has also been successfully used for the recovery of valuable compounds from OMWs. Previously, Paraskeva et al. (2007) compared the performance of NF

Table 11.5 Integrated membrane system for the recovery of phenolic from artichoke wastewaters (Conidi et al. 2014)

Technique employed	Process carried out	Recovered bioactives	Results and concluding remarks
UF coupled with two different NF Natural source: Artichoke wastewaters Three stage process	During the first stage, waste waters were ultrafiltered. In the second stage process, the raw were clarified using poly ether sulfone NF membrane NP030(400 Da) and followed by a cross linked aromatic polyamide NF membrane Desal DL (150-300 Da).	Phenolic compounds analysed (chlorogenic acid, cynarin and apigenin-7-O-glucoside)	The UF treatment preserved the phenolic bio actives such as chlorogenic acid, apigenin-7-0-glucoside and cynarin, TAA and sugars such as glucose, fructose etc. in the clarified stream due to the low rejection measured in the range of 1.2-8.6% while suspended solids were 100% completely retained in the retentate. The performance of both the NF membranes was compared. They showed high retentivity towards phenolic compounds in the range of 82-96%.: The permeate from the first NF membrane was deprived of sugars due to the high rejection values obtained and they were recovered in the permeate stream of the second NF membrane in which the rejection was in the range 3.4-5.5%.

and RO membranes in the concentration of pre-treated OMWs. Raw wastewaters were pre-filtered with a Polypropylene membrane (pore size of 80 μm) and treated via multichannel ceramic membranes having the pore size of 100 nm. This step produced a rejection of high MW constituents including fats, lipids and suspended particles. The permeate was then processed by using two different NF and RO polymeric spiral-wound membranes with MWCO of 200 and 100 Da, respectively. The concentrate contains more than 95% of phenolic compounds; however, better efficiency was achieved by applying RO.

In addition to the varied attempts in the recovery of bioactive through an integrated process, evaluation of its potential based on the use of membrane technology and adsorbent resins for the recovery, concentration and purification of phenolic compounds from artichoke wastewaters as a novel approach (Conidi et al. 2015) (Fig. 11.18, Table 11.9).

NF experiments were conducted extensively by Giacobbo et al. (2013), who used five different membranes for the fractionation of a winery effluent and recovery of the polysaccharides of low molecular weight and polyphenols. Three laboratory-made cellulose acetate membranes and two commercial membranes used: Evaluation was based on the analysis of rejection coefficients to polysaccharides, polyphenols, conductivity and total organic carbon. The rejection coefficients of polyphenols were overall lower than the ones of polysaccharides, showing that the polyphenols

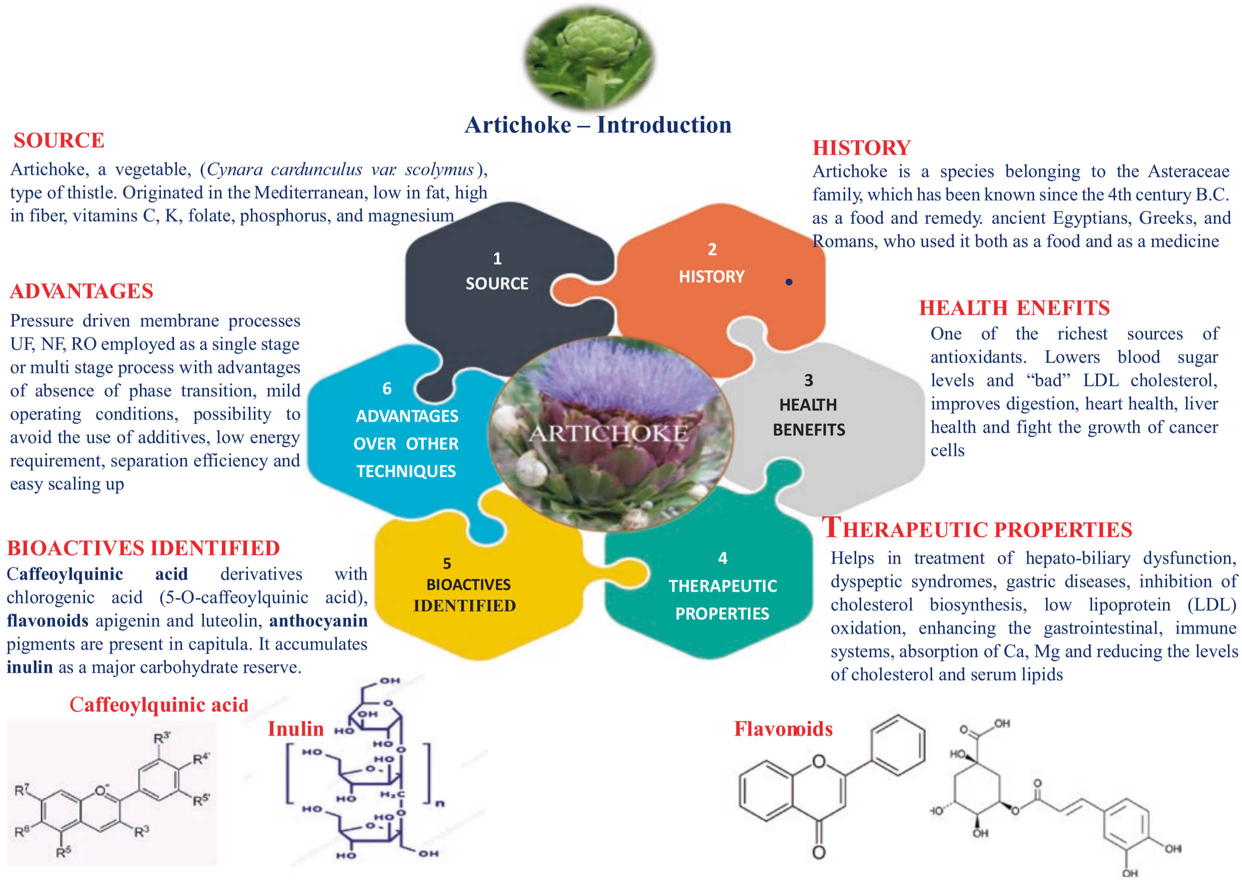

Fig. 11.7 Background on advantages, bioactive compounds and related health benefits in Artichoke

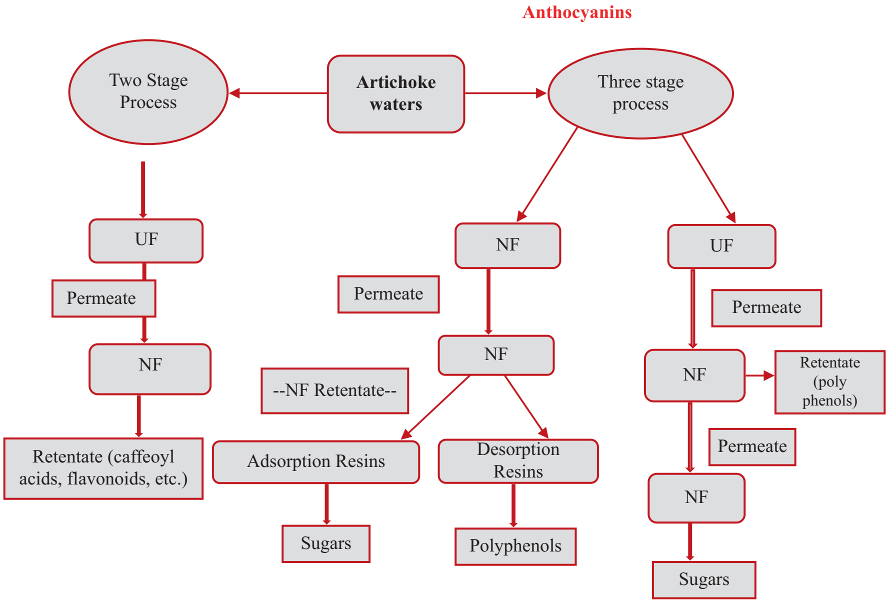

Fig. 11.8 Strategy description for the recovery of phenolic from artichoke wastewaters (Conidi et al. 2014)

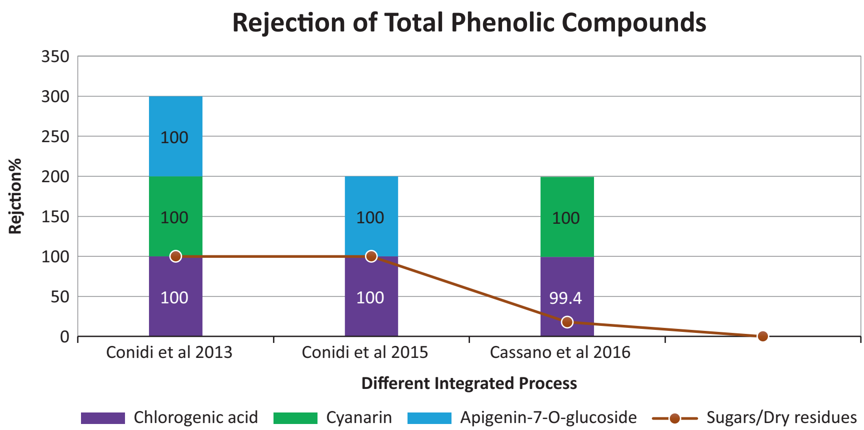

Fig. 11.9 Analytical studies and comparison among different studies reported by Cassano's group

permeate preferentially through all the membranes. The highest rejection coefficients were observed by the usage of NF270 membrane in the order of 93.8% and 99% for polyphenols and polysaccharides, respectively and the ETNA01PP membrane displayed the lowest rejection coefficients of 27% to polyphenols and 72% to polysaccharides.

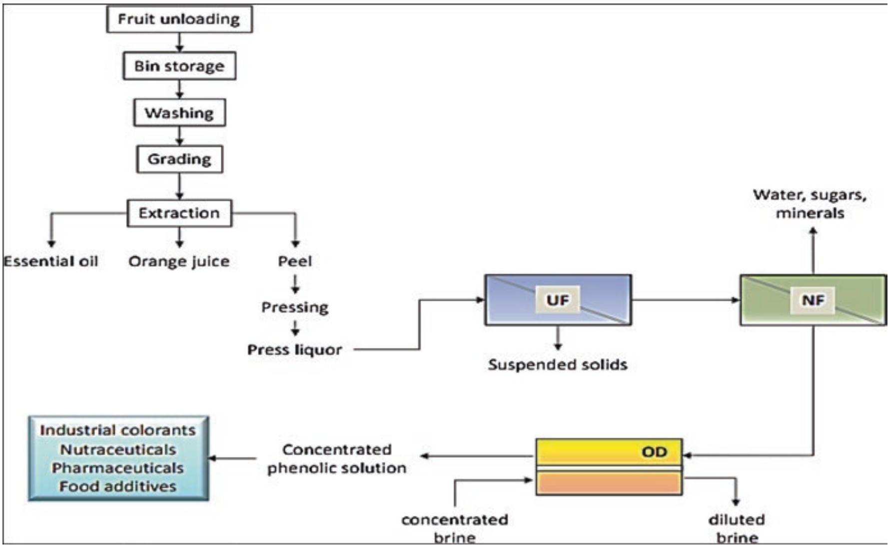

Fig. 11.10 Schematic of the integrated membrane process proposed for the recovery of flavonoids from orange press liquor (Cassano et al. 2014)

Another versatile integrated membrane process for the treatment of Nixtamalization wastewaters commonly known as Nejayote (Castro-Muñoz et al. 2015, 2017b; Ramírez-Jiménez and Castro-Muñoz 2020). It was carried out as a sequence of one MF pre-treatment step followed by two UF processes was investigated on a laboratory scale operating in selected process conditions (Castro-Muñoz and Yáñez-Fernández 2015) (Table 11.10).

Onsekizoglu (2013) demonstrated the capacity of membrane integrated process by OD and coupled MD process than performance compared with the thermal evaporation process (Figs. 11.19, 11.20, and 11.21, Table 11.11).

Conidi et al. (2017) investigated the use of flat-sheet UF and NF membranes with MWCO ranging from 1 to 4 k Da for the recovery of phenolic compounds from clarified pomegranate juice (Figs. 11.22 and 11.23, Table 11.12).

2.3 Four Stage Integrated Process

One another interesting example of an integrated membrane process for the recovery, purification and concentration of polyphenols from OMWs was demonstrated by Garcia-Castello et al. (2010) (Figs. 11.24, 11.25, and 11.26, Table 11.13).

MF/UF and NF/RO membranes in a sequential design allowed the recovery of water and bioactive compounds from Olive Mill Wastewaters (OMWs) was demonstrated by Russo (2007) (Table 11.14).

Piacentini et al. (2016) designed an innovative process for water recovery and polyphenols encapsulation from olive mill wastewaters (OMWWs) (Table 11.15).

2.4 Five Stage Integrated Process

Alberto et al. (2019) established that the wine lees extracted from wastewaters during the winemaking process possess a high concentration of bioactive molecules that can be used to obtain extracts or semi-finished products for food, nutraceutical and pharmaceutical applications. This was facilitated by means of integrating the new emerging technologies such micro wave assisted extraction with the pressure-driven membrane-based operations, for the recovery of phenolic compounds (Table 11.16).

Tamires Vitor Pereira et al. (2020) integrated the pressurized liquid extraction technique with NF and sequential MF-NF processes in the cross-flow filtration system cross and investigated the bioactive extraction and reported that it is an efficient method for the recovery and concentration of bioactive compounds from grape marc and a promising technique for obtaining functional products with high added value (Table 11.17).

Investigations performed on single membrane techniques so far establish that MF, UF, NF have been able to perform a primary purification of plant resources and microorganism sources due to their intrinsic properties (high efficiency, simple equipment, convenient operation and low energy consumption). These techniques can however be used in conjunction with other separation processes, to achieve various higher levels of fractionation. Further research is still going on to improve the inherent high throughout characteristics to convert the membrane-based techniques as the best productivity tools for the purification of bioactives.

Bagci et al. (2019) in their recent work, investigated a very novel way by coupling the RO and OD process using a commercial thin-film composite (TFC) polyamide reverse osmosis (RO) membrane which is activated by surface modification using a low-pressure nitrogen plasma.

A remarkable increase in water flux of the LPNP modified RO membrane with increased hydrophilicity was observed throughout the RO process. Substantial increase of higher soluble solids content (SSC) values in the concentrated juice at the same period of time rather than an untreated RO membrane. It also enabled 30% time-saving during the further osmotic distillation process.

2.5 Lactoferrin (Lf) isolation – Integrated Membrane Systems

Lu et al. (2007) isolated LF from bovine colostrum using UF followed by purification with a fast flow strong cation exchange chromatography system on a production scale (Figs. 11.27 and 11.28, Table 11.18).

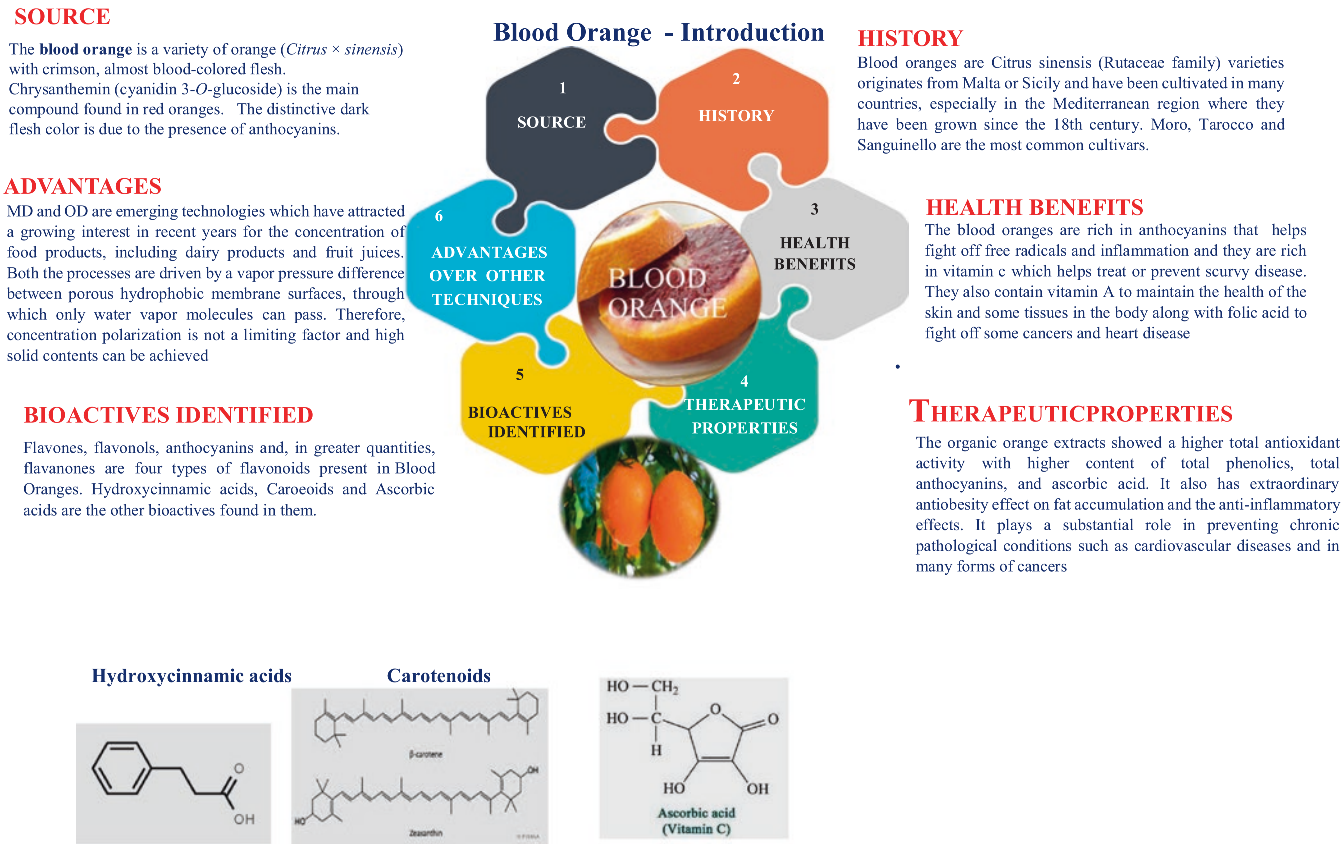

Fig. 11.11 Background on advantages, bioactive compounds and related health benefits in blood orange

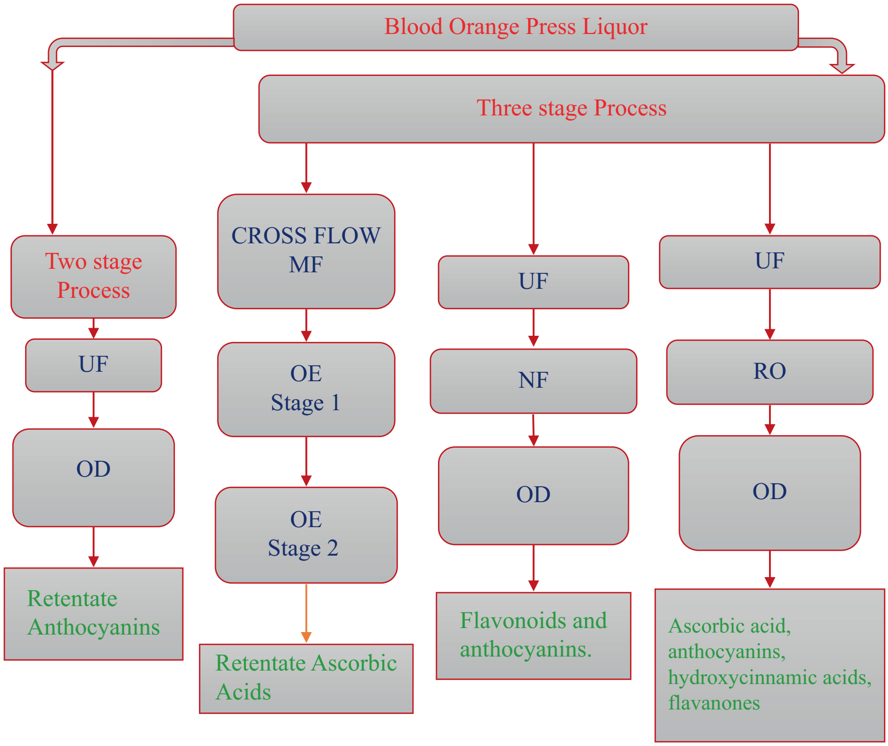

Fig. 11.12 Extraction of Bioactive - Integrated Membrane process in blood orange press liquor (Cassano et al. 2014)

The separation of high-value minor protein lactoferrin from crude dairy streams is a great challenge for the dairy industry. Wang et al. (Wang et al. 2020) investigated an electrodialysis with a UF filtration membrane (EDFM) approach to separate lactoferrin (LF) and immunoglobulins (Ig) from other dairy proteins (Table 11.19).

Brisson et al. (2007) superimposed an electrical field to a conventional membrane filtration unit and established that electrically-enhanced membrane filtration (EMF) increased the selectivity of LF separation in a mixed solution using a whey protein isolate (WPI) as a model (Table 11.20).

3 Conclusion and Future Prospects

Integration of various membrane technologies with better efficiency for bioactive component recovery will not only nurture the value for food waste but also reduce the cost of formulated products and thus minimize the use of synthetic chemicals in such formulations. An increasing amount of food and agriculture wastes are

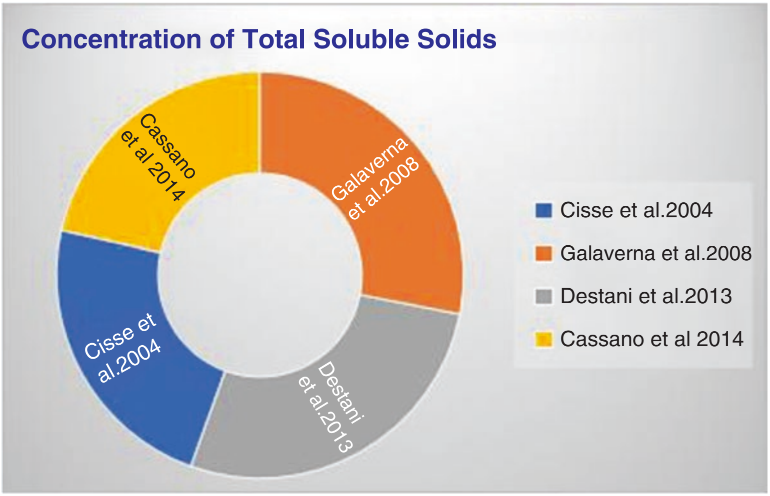

Fig. 11.13 Analytical studies and comparison among different studies reported Galaverna et al. (2008) based on the new integrated membrane technique produced a high-quality concentrated blood orange juice as an alternative to thermal evaporation

Table 11.6 Integrated membrane system for the recovery of anthocyanins from orange press liquors (Galaverna et al. 2008)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
UF coupled with RO followed by OD Natural source: Orange press liquor Three stage process	The process was based on the initial clarification of freshly squeezed juice by UF; the clarified juice was successively concentrated by two consecutive processes: First RO, used as a pre-concentration technique (up to 25–30^{0} Bx), then OD, up to a final concentration of about 60^{0}Bx	Ascorbic acid, anthocyanins, hydroxycinnamic acids, flavanones.	Integrated membrane process may be proposed as a very good alternative to obtain high quality concentrated juice, as end product also showed a very high antioxidant activity. Recovery of huge amount of natural bioactive components, showing a brilliant red colour and a pleasant aroma, characteristics that were predominantly lost when thermal evaporation was carried out.

Table 11.7 Integrated membrane system for the recovery of polyphenols from Bergamot juice

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
UF assisted two NF process Natural source: Bergamot juice Three stage process	The clarified juice was treated with a fluoropolymer UF membrane with a MWCO of 1 kDa (Etna 01PP, alfa Laval) The second step is the concentration of permeate in two different ceramic NF membranes (Inopor) with MWCO of 750 and 450 Da in order to evaluate the effect of the MWCO on the rejection of the membranes.	Polyphenols, flavonoids	The results indicated that the best separation of polyphenols from sugars occurred with the 450 Da membrane (higher rejection towards flavonoids and moderate rejection towards sugars). Permeate was a clear solution enriched in sugar and organic acids; The phenolic compounds were recovered on the retentate side, as also confirmed by high total antioxidant activity of the NF retentate

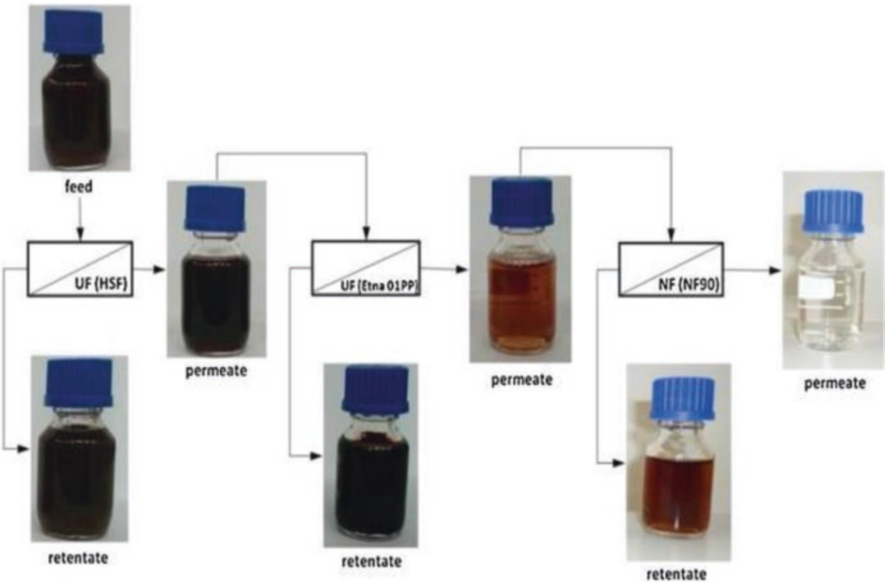

Fig. 11.14 Permeate and retentate samples obtained in the treatment of OMWs by integrated membrane operations (Cassano 2013)

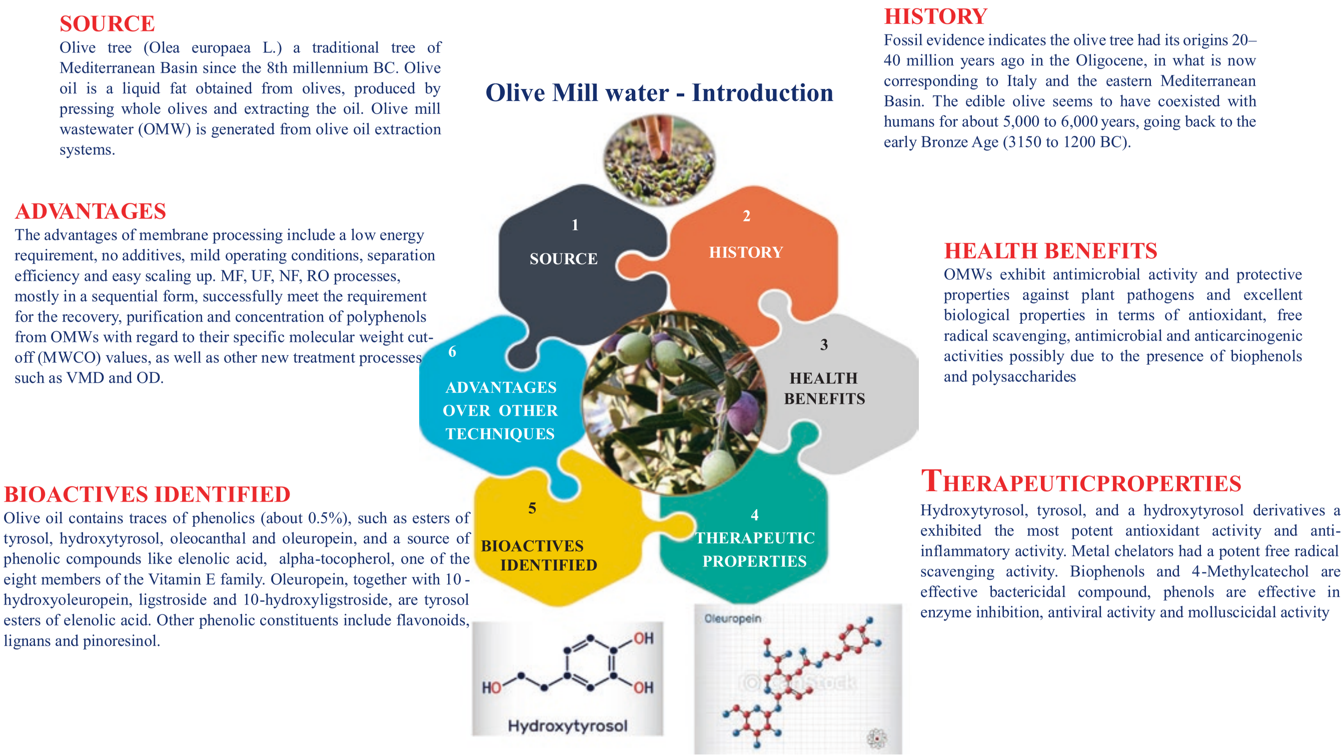

Fig. 11.15 Background on advantages, bioactive compounds and related health benefits in Olive Mill water

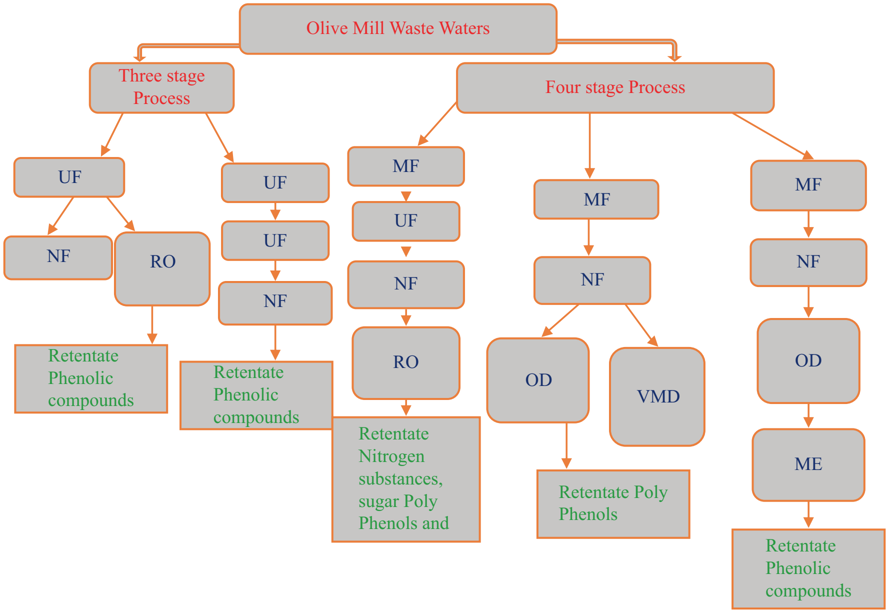

Fig. 11.16 Extraction of Bioactive - Integrated Membrane process in Olive Mill waste waters

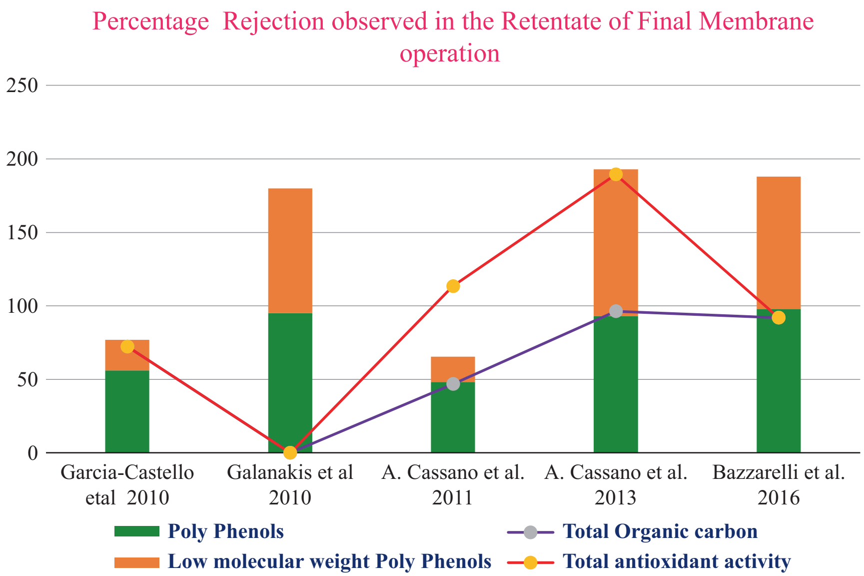

Fig. 11.17 Analytical studies and comparison among different studies reported

Table 11.8 Integrated membrane system for the recovery of polyphenols from Olive Mill waters

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
Two different UF steps coupled with NF membrane. Natural source: Olive Mill waste waters Three stage process	First UF step performed with hollow fibre membranes with a nominal pore size of 0.02. The UF permeate was then processed with a flat-sheet UF membrane (Etna 01PP, alfa Laval) with a MWCO of 1000 Da. The UF permeate form the second UF step concentrated by NF using a spiral-wound membrane (NF90, Filmtec/Dow)	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) was obtained	Ultrafiltration process ensured the complete removal of suspended solids and also showed low rejections towards low molecular weight polyphenols (2.1 and 17.6% respectively). The NF membrane retained all the analyzed phenolic compounds producing a permeate depleted in phenolic compounds. These recovered fractions were considered of interest for cosmetic, food and pharmaceutical industries as liquid, frozen, dried or lyophilized formulations.

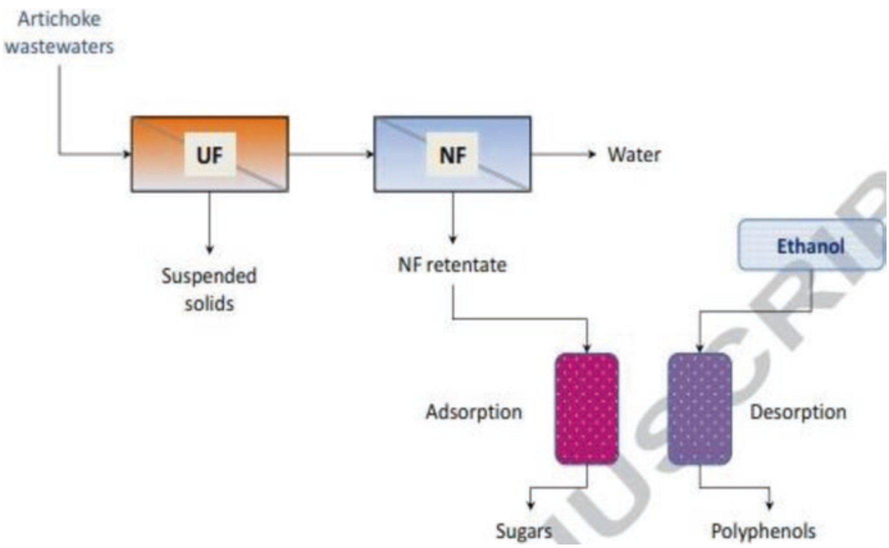

Fig. 11.18 General scheme of the investigated process (Conidi et al. 2015)

available from food processing industries and post-harvest losses of fruits and vegetables and their utilization as a source of nutritious bioactive compounds would certainly increase the financial status of farmers across the globe and decrease the burden of waste management (Kumar et al. 2017). Enhancement of

Table 11.9 Integrated membrane system for the recovery of phenolic compunds from Artichoke Waste waters

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
UF, NF and adsorption-desorption tests Natural source: Artichoke waste waters Three stage process	Initial step is pre-treatment by UF. The UF permeate was submitted to a NF process. Three different macroporous resins were tested through adsorption/desorption methods	Phenolic compounds, such as chlorogenic acid (CA) and apigenin 7-O-glucoside (AOG)	Suspended solids and macromolecular compounds were completely removed from the artichoke wastewaters by UF producing a permeate stream enriched in phenolic compounds and sugars. Phenolic compounds concentrated by NF with a production of a retentate stream containing about 1.6 g/L of CA and 0.3 g/L of AOG. Among the three different tested resins, the S 7968 offered the best performance in terms of adsorption/desorption ratio for CA, with a Total adsorption desorption yield (TADY) of 63.39%; while for the AOG the S 7968 and the S 2328 resins showed a TADY in the range 68.31-78.45%

membrane technology through an innovative approach in extraction technology with lesser or no use of solvent will be of great significance to achieve a sustainable bioprocess. Added to that, serious environmental issues related to the disposal of agricultural and industrial wastes in the current scenario in our country and the rest of the world can very well be managed and subsequently by the rational use of the bioresources through the realization of suitable and appropriate membrane technologies coupled together. In this regard, a thorough analysis and assessment of the waste generated, complete utilization as nutraceuticals with many commercial ventures by setting up membrane operation units would help to establish the usage of bioactive residues to eliminate environmental implications in the future. The functional properties of the compounds are preserved since the product is not subjected to high temperatures and no physical changes in the solvent and the process itself is energy saving. Due to the ever-increasing demand and cost of energy, integrated membrane processes are inevitable which are gaining prominence in food processing and they will be widely used by the food and allied industries in the years to come.

Table 11.10 Integrated membrane system for the recovery of poly phenols from Nixtamalization wastewaters

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
MF followed by two UF process Natural source: Nixtamalization wastewaters Three stage process	Nejayote was submitted to a preliminary MF process Permeate obtained from MF was submitted to a UF step. The UF unit was equipped with two different polysulfone hollow fiber membranes	Poly phenols	1. MF retentate contains concentrated solution rich in suspended solids, parts of the grain and macromolecules that could be used as feed on livestock or can be used as carbon source to a biotechnological process in order to produce biogas, bioethanol or other type of biotechnological products. 2. UF retentate (100 kDa) concentrated fractions can be used to food additives. 3. UF retentate (1 kDa) concentrated fractions containing components of soluble calcium can be reused in the Nixtamalization process of maize. 4. The clear permeate (1 kDa UF) enriched in polyphenols could be used in cosmetic, food, and pharmaceutical industries as well as liquid formulations after fractionation by NF membranes or concentration by reverse osmosis. 5. In addition, the process of treatment of Nejayote by membrane process avoids water pollution and provides an environmental solution to decrease the biochemical oxygen demand.

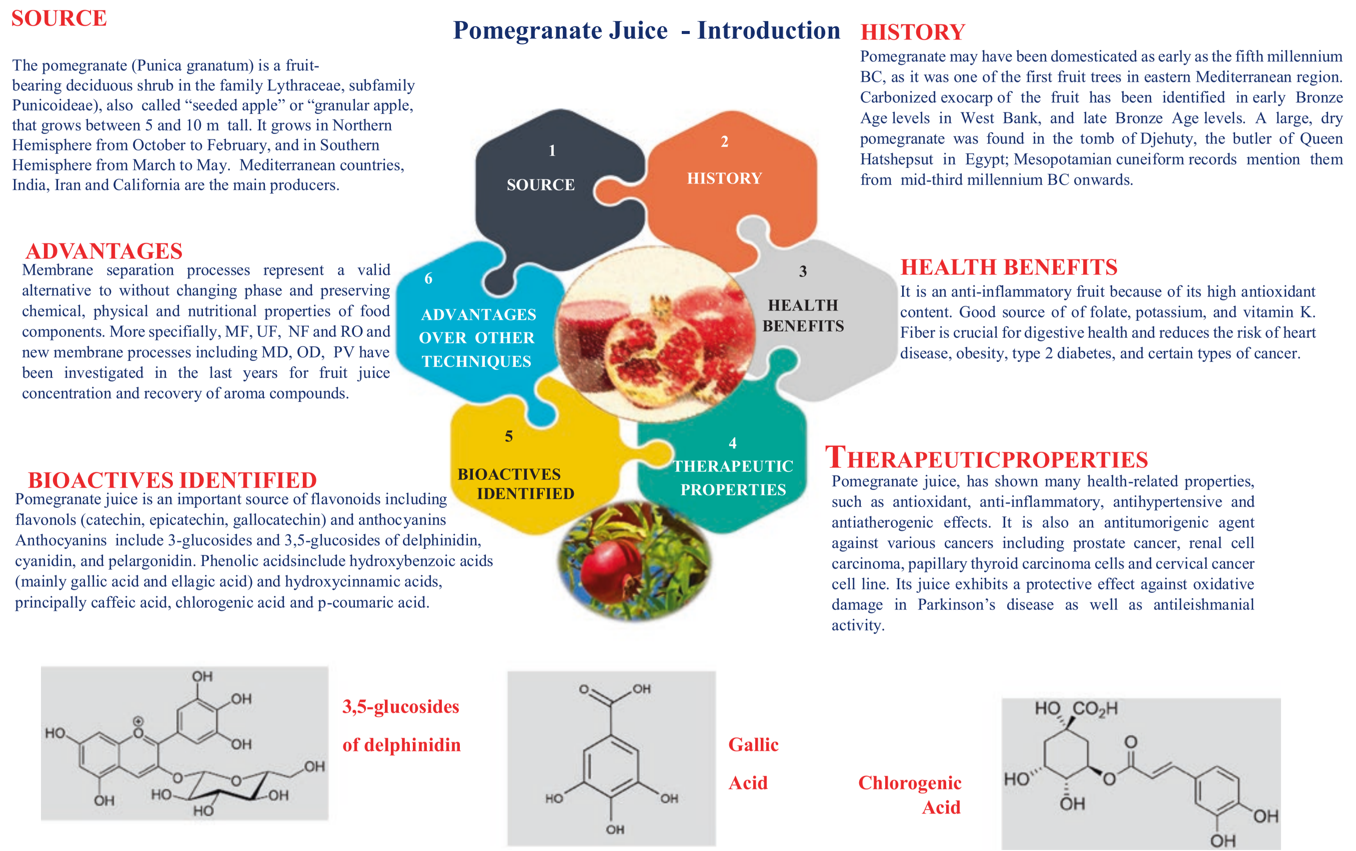

Fig. 11.19 Background on advantages, bioactive compounds and related health benefits in Pomegranate Juice

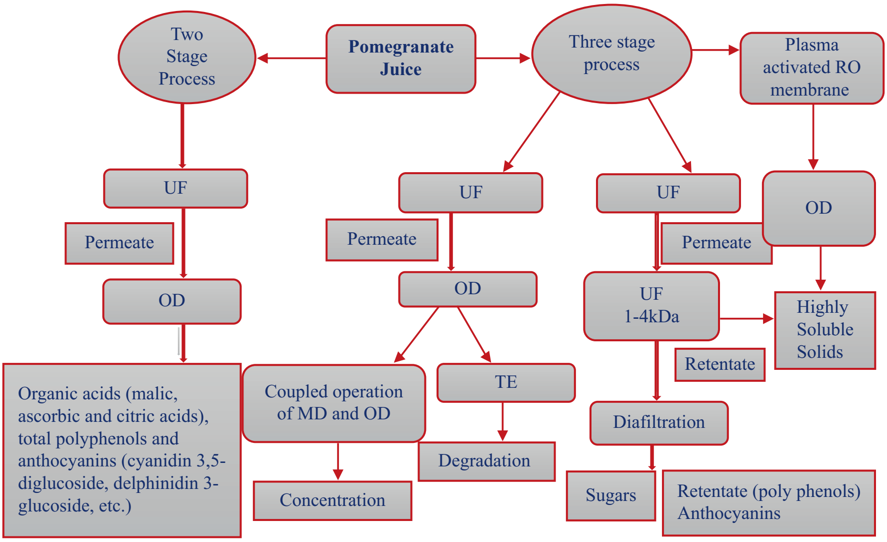

Fig. 11.20 Extraction of Bioactive - Integrated Membrane process in Pomegranate Juice

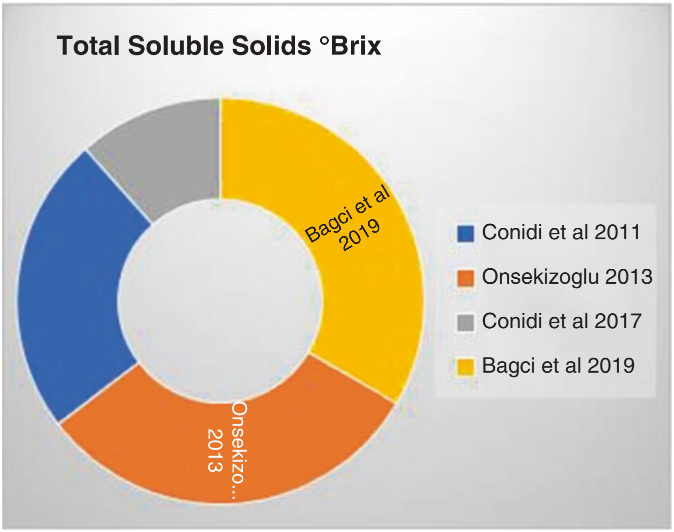

Fig. 11.21 Analytical studies and comparison among different studies reported

Table 11.11 Integrated membrane system for the recovery of Anthocyanins from Pomegranate juice

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
UF, OD and coupled membrane distillation (CO) Natural source: Pomegranate juice Three stage process	Initial step is pre-treatment by UF. The clarified juice was subsequently submitted to a concentration procedure using both membrane-based processes (OD and coupled membrane distillation) and traditional thermal evaporation (TE).	Hydrolyzable tannins, anthocyanins and ellagic acid derivatives	The performance of ultrafiltration system was improved by the pre-clarification of pomegranate juice by lesser amounts of gelatin and bentonite, in comparison with conventional applications. The clarified pomegranate juice was concentrated up to 55.5 ° Bx in 390 min with OD, the CO yielded a concentration of the clarified pomegranate juice up to a value of 57.4^{0}Bx in 240 min (Fig. 11.4), confirming enhancement of process performance. The approach for coupled operation of MD and OD concept was more promising for concentration of pomegranate juice, allowing higher concentration to be reached in shorter periods of operation with a slight increase in juice temperature.

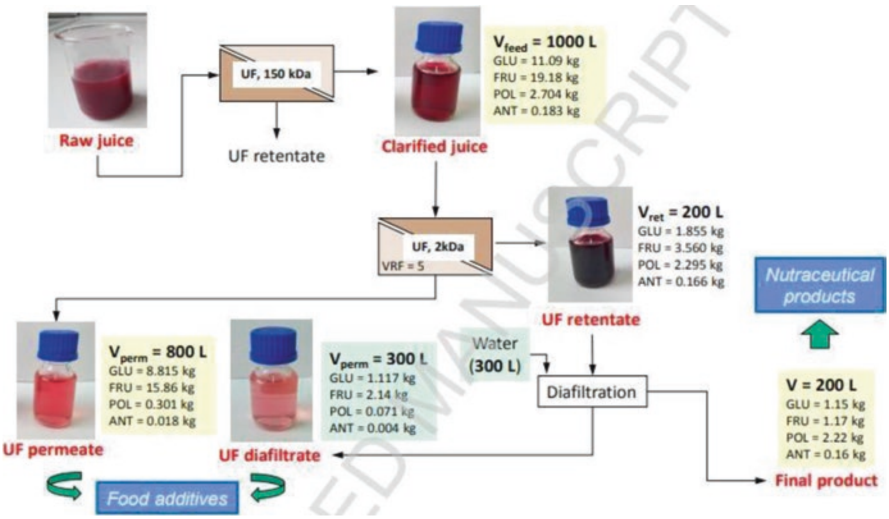

Fig. 11.22 Mass balance of the fractionation process of the pomegranate clarified juice with Desal GK membrane (Conidi 2017)

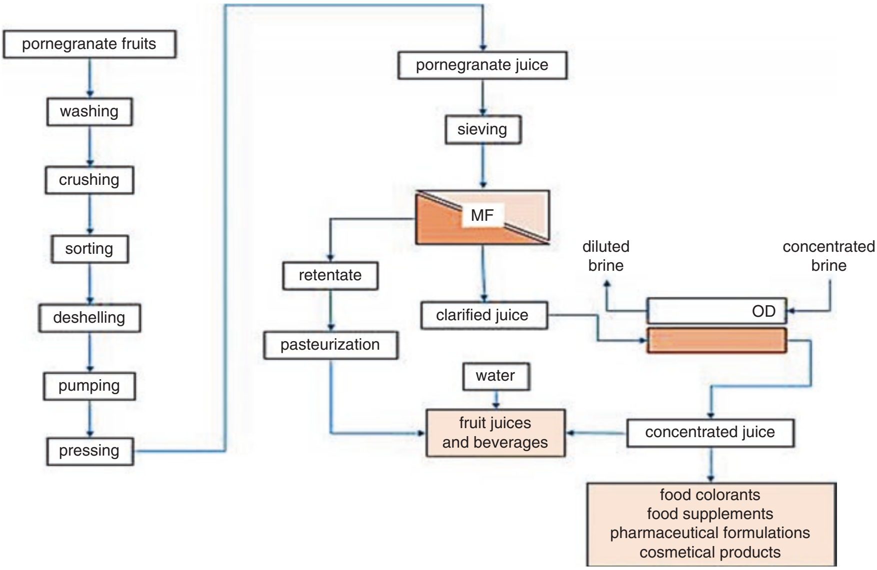

Fig. 11.23 Integrated membrane process for the production of high-quality concentrated pomegranate juice (Conidi 2020)

Fig. 11.24 -Flowchart representing the activities carried out for the recovery, purification and concentration of polyphenols from olive mill wastewaters. (Garcia-Castello et al. 2010)

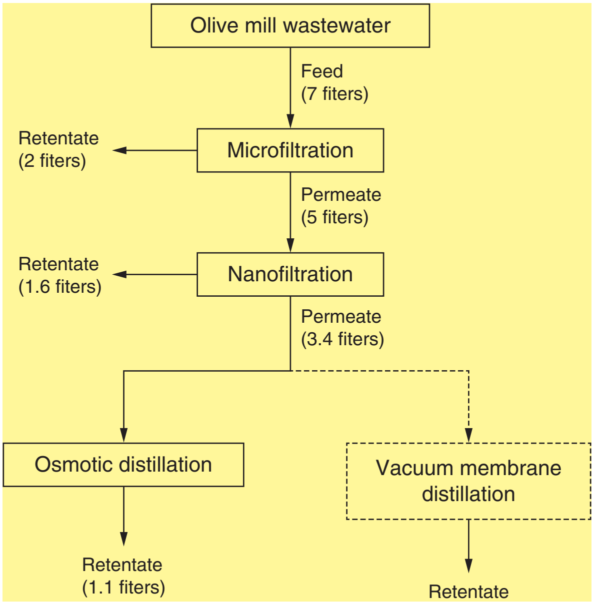

Table 11.12 Integrated membrane system for the recovery of Polyphenols from Pomegranate juice(Conidi et al. 2017)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
(UF)coupled with (NF) Natural source: Pomegranate juice Three stage process	Raw juice was clarified using UF membranes In the second step, the permeate was treated with four different flat-sheet UF membranes having a MWCO ranging from 1-4 kDa Retentate stream was concentrated via diafiltration step.	Polyphenols and anthocyanins	From the observed results, mass balance of the membrane fractionation process was carried out in order to quantify the amount of bioactives and sugars recovered in the varied permeate and retentate samples collected. In the 200 L of final concentrated solution obtained, yield of polyphenols and anthocyanins in the retentate stream are of the order of 84.8% and 90.7%, respectively. After diafiltration, efficiency of glucose and fructose recovery can be increased up to 90% and 93%, respectively. Final retentate with very high antioxidant activity can be reused for the formulation of nutraceutical products or as a natural colorant in alternative to the use of synthetic substances; Residual permeate and diafiltrate streams, containing high content of sugars, can be reused as food additives or as bases for soft drinks.

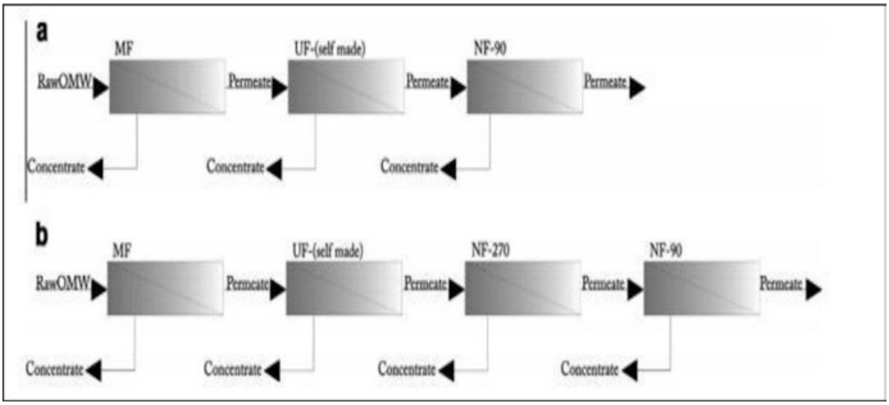

Fig. 11.25 Schematic of two different IMS arrangement used (Zirehpour et al. 2012)

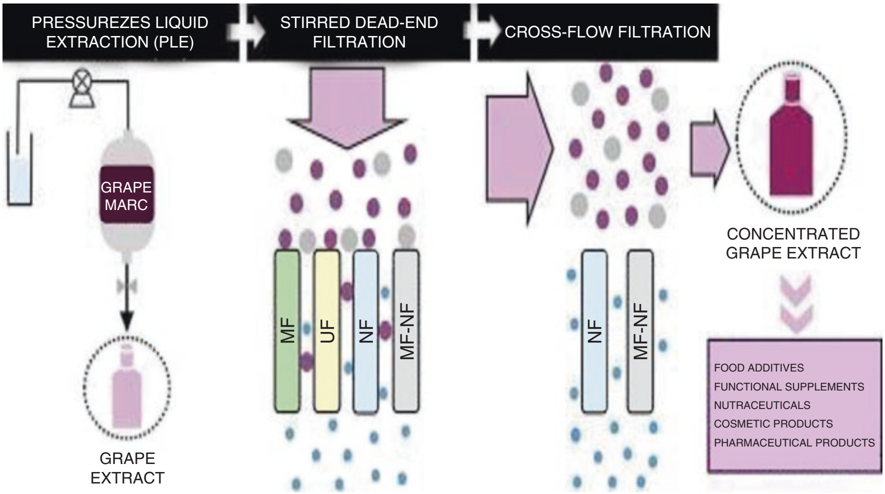

Fig. 11.26 Schematic of the integrated membrane process proposed for the recovery of bioactive from grape marc (Tamires Vitor et al. 2020)

Table 11.13 Integrated membrane system for the recovery of Polyphenols from Olive Mill wastewaters(Garcia-Castello et al. 2010)

Technique employed	Process carried out	Bioactives recovered	Results and Concluding remarks
UF coupled with NF followed by osmotic and vacuum distillation Natural source: Olive mill waters Three stage process	The olive mill waters were first clarified using by using a tubular Al_2O_3 MF membrane with a pore size of 200 nm. Sample was treated with a NF spiral-wound membrane. In the third step, the permeate was separated by OD using PP hollow-fibre membrane module and vacuum membrane distillation (VMD)	Poly phenols	Permeate from MF membrane contained 78% of phenolic derivatives, total amount of suspended solids 91% and TOC reduced to 26%. The NF treatment step lead to further reduction of TOC from 15 g/L to 5.6 g/L with concentration of low molecular weight polyphenols in NF permeate with record of low rejection observed from 1% to 21%. The concentrated solution of OD contained about 0.5 g/L of free low molecular weight polyphenols.

Table 11.14 Integrated membrane system for the recovery of Polyphenols from olive Mill waters

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
MF coupled with UF with NF/RO Natural source: Olive mill wastewaters Four stage process	Initial MF step, was performed by ceramic membranes of 0.8 and 0.45 μm and a polymeric spiral-wound membrane 500 kDa. In the second step, the permeate was treated with a flat-sheet UF membrane having a MWCO in the range of 1-80 kDa Permeate stream was concentrated with a NF membrane. Finally, RO concentrates all the components of UF permeate.	Low molecular weight free polyphenols (hydroxyl-tyrosol, and oleuropein.	The content of free low molecular weight polyphenols in the untreated OMWs was lower (55.38 mg/L than that measured in the MF permeate (349.18 mg/L). For free polyphenols, the UF 6 kDa membrane showed a rejection of about 45%. Rejections for oleuropein and hydroxytyrosol were 75% and 45%, respectively. Effect of a nanofiltration step (NF) on UF permeate, in terms of selectivity, purification and yield of hydroxytyrosol, is not reported. RO shows a rejection of 99.9% respect to nitrogen substances, sugars and polyphenols and between 83% and 99% respect the ionic species.

Table 11.15 Integrated membrane system for the recovery of Polyphenols from olive Mill water (Piacentini et al. 2016)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
MF and NF coupled with OD and membrane emulsification (ME) Natural source: Olive mill waters Four stage process	First step, olive mill waters was treated by MF process to remove suspended solids. Then OMWWs were processed by NF in order to obtain water from the permeate side and a concentrated polyphenolic solution from retentate side. NF retentate was passed through OD to remove water and concentrated polyphenolic stream was encapsulated in a water-in-oil emulsion by ME.	Poly phenols	Concentration of polyphenolic solution from retentate of OD produced very high fraction of low molecular weight polyphenols according to a concentration factor of 7. This fraction which is formulated by the ME process resulted in the encapsulation efficiency of 90%. According to this process mass balance, 1000 L of treated OMWWs, fractionated 1463 g of phenolic compounds (85% of the initial phenolic content) and recovered 800 L (80% of the initial volume) of purified water.

Table 11.16 Integrated membrane system for the recovery of Phenolics from wine lees (Alberto et al. 2019)

Technique employed	Process carried out	Bioactives recovered	Results and concluding remarks
Microwave assisted extraction coupled with MF and processed by UF and two different nanofiltration process Natural source: Wine lees Five stage process	First step, wine lees was subjected to different microwave power (range: 90–350 W) and exposure time (range: 0.5 − 3 min). Total of 12 replicated trials were carried out. The red wine lees extract was previously microfiltered by using a polyvinylidene fluoride (PVDF) membrane with a membrane pore size of 0.15 m. Then the clarified solution from MF was treated by using three different flat-sheet membranes (one UF and two NF membranes) with a molecular weight cut off in the range 150–1000 Da.	Phenolics, proanthocyanidins	The retention of the MF membrane towards proanthocyanidins resulted of about 42%; Combination of MF and NF, an attractive alternative process for producing, at low temperatures, preconcentrated extracts from wine lees without thermal damage before final concentration (vacuum evaporation, osmotic evaporation) or spray drying. Hydro-alcoholic extracts were clarified by microfiltration. Among the selected membrane, the ETNA 01PP exhibited the highest productivity in selected operating conditions but lower retention of phenolic compounds and sugars in comparison with the other membranes. On the other hand, the NFT-50 membrane presented retention coefficients higher than 70% for all detected free low molecular weight phenolics.

Table 11.17 Integrated membrane system for the recovery of Phenolics from grape Marc (Tamires Vitor et al. 2020)

Technique employed	Process carried out	Recovered bioactives	Results and concluding remarks
Pressurised liquid extraction coupled with dead end and cross flow NF and sequential MF-NF processes Natural source: Grape Marc Multi stage process	First step, four NF membranes were tested in terms of permeate mass flux and retention index of total monomeric anthocyanins and total phenolics. Second step, the use of MF and UF processes were evaluated as alternatives to improve the concentration performance and reduce membrane fouling in the NF step. After the preliminary tests in stirred dead-end module, selection of membranes, a cross-flow filtration system was used. NF and sequential MF-NF processes were investigated in the cross-flow filtration system Usage of non-toxic solvent (water–- ethanol pH 2.0 (50% w/w)) at moderate temperature (40 °C) under high pressure (10 MPa)	Phenolics, anthocyanidins	Highest retention of antioxidant capacity (52%) was observed with MF-NF sequential process. The maximum retentions of monomeric anthocyanins were obtained with NP030 (98%), followed by NF270, NP010, and MV020-NP010 membranes, with 84.0, 78.6, and 78.2%, respectively. Added to that, NP030 membrane also provided a very high retention value for total phenolics (91%), whereas NP010, NF270, and MV020-NP010 achieved values of 79%, 74%, and 71%, respectively. Moreover, NP030 membrane obtained the highest retention of total solids (43%), and the MV020-NP010 process achieved the lowest value (5%). Due to the previous treatment with the MV020 membrane, the concentrated extract obtained from the sequential process MV020-NP010 is more purified, mainly in terms of the total solids content.

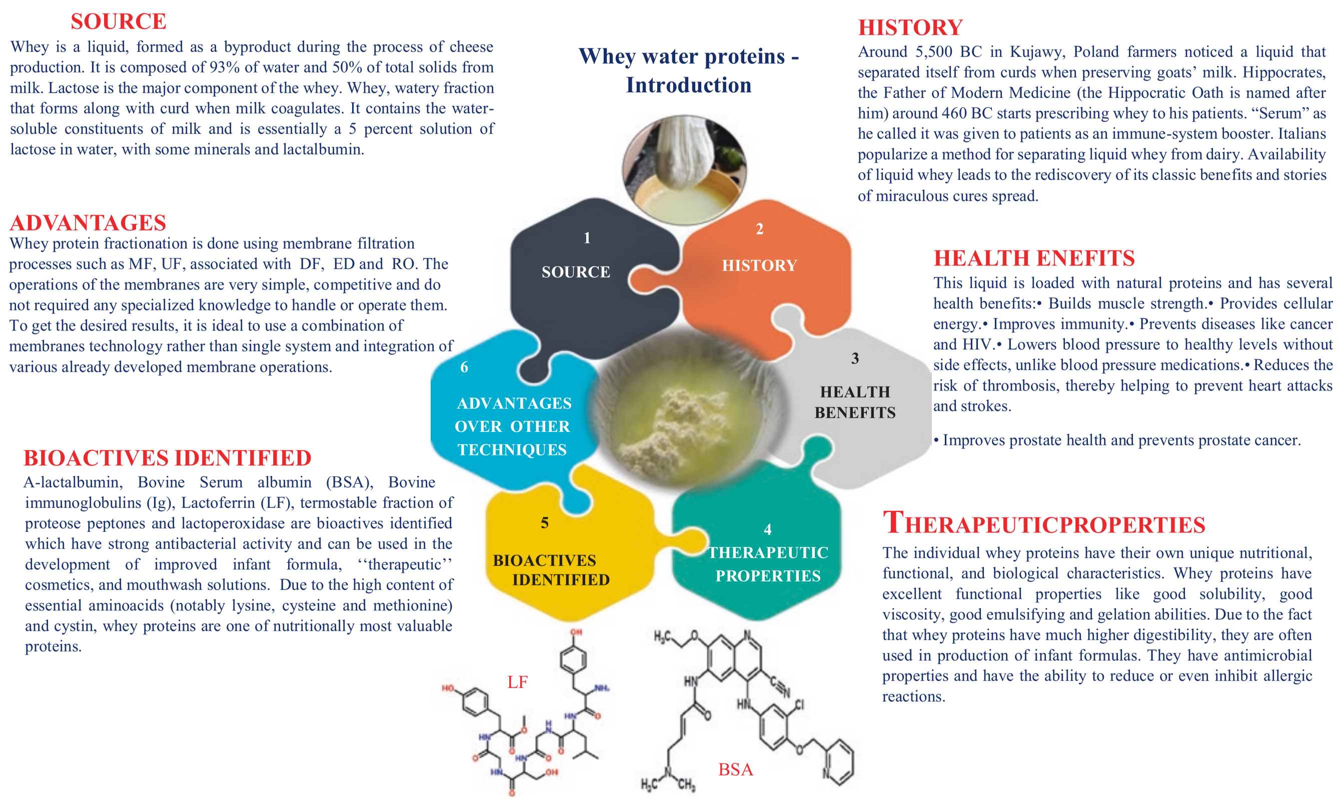

Fig. 11.27 Background on advantages, bioactive compounds and related health benefits in Whey water Proteins

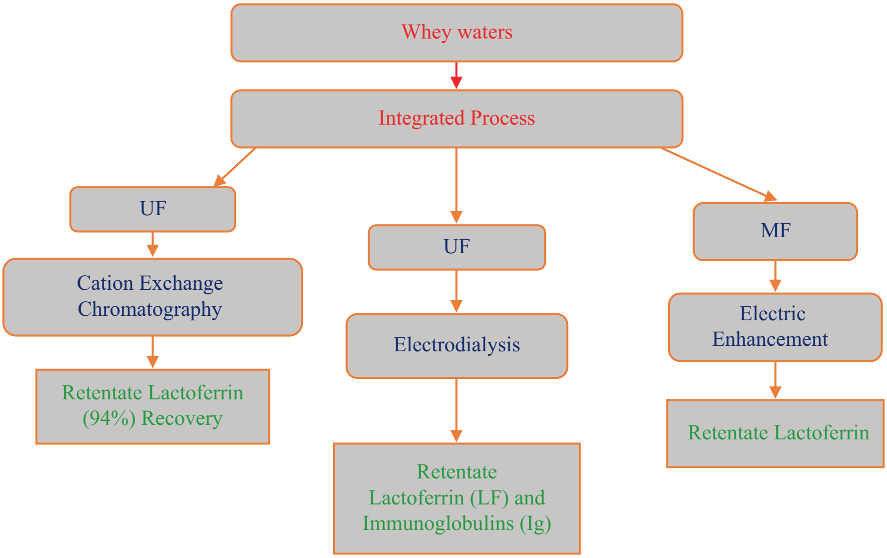

Fig. 11.28 Extraction of Bioactive – integrated Membrane process in Whey waters

Table 11.18 Integrated membrane system for the recovery of lactoferrin from Bovine Skim Colostrum Whey (Lu et al. 2007)

Technique employed	Process carried out	Bio actives recovered	Results and concluding remarks
Separation and concentration of LF by UF followed by purification of the crude LF using fast flow cation exchange chromatography Natural source: Bovine skim colostrum whey Two stage process	Two-step UF process was performed with membranes of nominal molecular weight cut-offs of 100 kDa for UF-1 and 10 kDa in UF-2. UF-1 performed at fixed transmembrane pressure (TMP), Tangential flow velocity and temperature equivalent to 200 kPa, 5 m/s, 25 °C and UF-2 at 4 m/s, 150 kPa, 50 °C. A stepwise procedure for purification of the crude LF was conducted using strong cation exchange chromatography	Lactoferrin (LF)	LF concentrated in the UF-2 retentate reached a purity of 30.88% (w/w) and a recovery of 94.04%. By using a strong cation exchanger, the purity and recovery of the final LF product was increased to 94.20% and 82.46% suitable for pharmaceutical applications. Low-temperature with no side reactions or additives makes it further acceptable as product of consumer quality

Table 11.19 Integrated membrane system for the recovery of lactoferrin from crude dairy streams (Wang et al. 2020)

Technique employed	Process carried out	Recovered bioactives	Results and concluding remarks
Electrodialysis with filtration membrane (EDFM) Natural source: Crude dairy whey streams. Novel in house coupled process	The EDFM comprises of polyvinyl alcohol (PVA) prepared by phase inversion in a coagulation bath with 80% ethanol to serve as the filtration membrane. Also, the setup contains two restriction membranes (polyacrylamide (PAm) with MWCO of 5 kDa and a pair of electrodes. The feed solution passes on one side of the membrane, with a buffer solution passing on the permeate side. Restriction membranes are used to allow the passage of ions to conduct the electric current, while preventing the proteins from entering into the electrode compartments. Two electric field strengths (38.5 and 77 V/cm) were then investigated within the electrical cell.	Lactoferrin (LF) and immunoglobulins (Ig)	Filtration membrane prepared in-house offered strong rejection for LF and Ig while allowing a high flux of other proteins. EDUF processes are found to be highly effective in separating large molecules with biological activity and uses no solvents or other chemicals that can add to the environmental footprint of the operation.

Table 11.20 Integrated membrane system for the recovery of lactoferrin from whey protein isolate (Brisson et al. 2007)

Technique employed	Process carried out	Recovered bioactives	Results and concluding remarks
Electrically enhanced membrane filtration using the conventional MF membranes. Natural source: Whey protein isolate Integrated process	Electrically-enhanced membrane filtration (EMF) process were performed on a purpose-built flat-sheet module with low transmembrane pressure (0.7 × 105 pa) and feed velocity (0.05 m s − 1). Electrical field acts as additional driving force to the transmembrane pressure (PT).	Lactoferrin (LF)	Differences in protein electrophoretic mobility are coupled to the membrane sieving effect to enhance the selectivity of membrane fractionation in EMF, also to improve protein permeation flux (JP) by preventing concentration polarization and membrane fouling. The iron-binding properties of LF have been used to modify its transmission coefficient behaviour in EMF in presence of whey proteins and improve the separation selectivity. The potential of EMF for separation of more complex protein mixture such as cheese whey LF is evident.

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