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REVIEW ARTICLE

Recovery of Bioactive compounds from food Processing wastewaters by Ultra and Nanofiltration: A review

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ABSTRACT
The recovery of bioactive compounds with high added value from by-products of food processing industry is a current challenge of research areas. Many food wastewaters have been treated with conventional membrane processes as Ultrafiltration (UF) and Nanofiltration (NF) in order to recover and concentrate different bioactives compounds in aqueous solution. The goal of the current article is to show and discuss briefly the main food processing aqueous systems that have been treated by membranes technologies. In addition, the paper shows the most recovered high-added value components in recent years.
Keywords: *Wastewaters, organic load, recovery, membrane technologies, high added value compounds.*

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Abbreviations:
MF: Microfiltration; UF: Ultrafiltration; NF: Nanofiltration; TMP: Transmembrane pressure; PSU: Polysulfone; PES: Polyethersulfone; PVDF: Polyvinyliden- fluoride; MWCO: Molecular weight cut-off; OMW: Olive mill wastewater; COD: Chemical oxygen demand; DOC: Dissolved organic carbon; TOC: Total organic carbon.

INTRODUCTION

Longtime, different membrane technologies has been used in order to clarify many fruit juices and large variety of new products, based on clarified fruit juices, have appeared in the markets these products can be achieved only by completely removing all suspended solids that is related to high turbidity in the juices [1,2]. Membrane processes as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) have been widely applied to the diary, food and beverage industry in order to reduce the storage and shipping costs, and to achieve longer storage in food products, by the way the membrane processes have been widely used due to their characteristics that represents great advantages during the processing [3,4].

Last decade, food industry has had several problems in terms of processing, obtaining new products with high added value and disposal of waste from food production processes. Recently, proposal methods based on membrane technologies have been used on treatment of wastewater and aqueous food systems [5]. Different methods to obtain purified extracts containing biologically compounds from fruit or vegetable products have been evaluated including solvent extraction, irradiation assisted extraction, hot water extraction, resin- based extraction, ultrasound-assisted extraction, enzyme-assisted extraction and supercritical fluid extraction [6,7,8,9]; physico-chemical methods that regularly get low yield and considerable losses. On the other hand, membrane technologies have been successfully employed for the purification and concentration of bioactive compounds from natural products with possible applications on food colorants, food supplements, pharmaceutical formulations and cosmetical products [10,11]. Specifically MF, UF and NF offer particular advantages in terms of absence of phase transition, mild operating conditions, low energy requirement, separation efficiency and easy scaling up when compared with conventional methodologies [12,13,14,15].

MF technologies are considered as macroscopic pre-treatment process, UF technologies are considered as media of macro and micro-molecules separation and finally the NF process are taken into consideration as isolation and purification technics, all these processes are named as conventional technologies and it consolidated on the recovery of high-added value components. In case of UF and NF processes are considered as membrane process with high selectivity in order to recover specific compounds such as volatile compounds, low molecular weight [16,17].
In general, this paper will provide a broad overview of the recent significant applications of the membrane process (MF, UF and NF) on recovery of bioactive compounds from food processing wastewaters.

CONVENTIONAL MEMBRANE PROCESSES: ULTRAFILTRATION AND NANOFILTRATION.
The membrane processes as Ultrafiltration and Nanofiltration are part of physicochemical and non-destructive techniques applied in different steps of the above downstream processing. Recently, researchers are focusing on recovery macro and micromolecules from different sources of food processing industry using UF and NF. Commercial membranes of UF going to pore sizes of 300 to 1 kDa, in case of 2-1 kDa membranes are considered as UF membranes as well as to the border of Nanofiltration systems for their narrow pore size [18]. However, UF processes have been successfully developed for recovering from macromolecules to micromolecules with low molecular weight from different food wastewaters such as dietary fibers, proteins, sugars, phenolic compounds and antioxidant components.
On the other hand, nanofiltration membranes are capable to recover, fractionate and concentrate different types of bioactive compounds with low molecular weight. Salehi [19] reported commercial membranes of NF from 500 Da up to 1000 Da; however, there are reports where membranes with lower molecular weight have been used in recovery of bioactive compounds. The membranes presents pore sizes from 120 to 450 Da [20, 21].
Finally, the separation performance of the UF and NF membranes is influenced by the operational parameters such as temperatures, transmembrane pressure and feed flow. But the chemical composition of the wastewater play an important role in the separation performance of the membrane due to their interactions between membrane surface and characteristics of the solutes [19]. However, taking into account all these factors, the recovery of bioactive compounds from food processing wastewaters has been successfully developed; brief general applications of the ultrafiltration and nanofiltration processes in this field are provided in the following section.
1.1. Ultrafiltration applications on recovery of bioactive compounds from effluents.
Membrane processes, including MF, UF, NF and integrated membrane processes have been also proposed to treat effluent streams from different wastewaters. The clarification of high-added value products (pectin containing solution and phenol containing beverage) from olive mill wastewaters (OMW) was investigated by Galanakis et al. [22], the extract was processed with four types of UF (100, 25, 10 and 2 kDa) membranes, the membranes of 25 and 100 kDa showed very satisfying results with regard to the concentration of pectin solutions as well as be able to separate them from cations phenols, the membrane of 25 kDa is considered a membrane with intermediate membrane process [18]; it was also able to remove partially the heavier fragments of hydroxycinnamic acid derivates and flavonols, and simultaneously to sustain the antioxidant properties of the phenol containing beverage in the permeate stream, the retention coefficients of the UF membranes were between 18-38 % on total sugars, 10-25% on total phenols and 10- 62% on flavonols.
In addition, polyphenolic and antioxidant compounds were recovered in the fractions produced by UF membranes from OMWs [14], in this study was used four different membranes by different MWCO (4, 5 and 10 kDa) and polymeric material (regenerated cellulose and polyethersulfone). It is important that these membranes have narrow membrane pore and they are nearby to the nanofiltration technology [18]. The regenerated cellulose membranes exhibited lower rejections (4.8-5.5%) on phenolic compounds, higher permeate fluxes and lower fouling index if compared with PES membranes. For the OMWs treatment, the UF process (100 kDa, area 0.0155 m^2, recycle flow rate 100-200 L/h, pressure 1-3 bar) has been demonstrated that have a considerable DOC (Dissolved organic carbon) removal efficiency around 60 to 80 % [23], the same author analyzed the effectiveness of the different UF membranes and operating conditions where it was concluded the retention coefficients on DOC (92.3%), TOC (Total organic carbon) (92.7%), suspended solids (97.1%) and oil-grease removal (98.9%). The highest permeate flux (25.9 L/m^2 h) was obtained using a MW membrane (Ultrafilic) under operational conditions of Q_f=200 L/h flow rate and TMP= 4 bar, while the highest removals were obtained at Q_f=100 L/h flow rate and TMP= 1 bar, the COD (chemical oxygen demand), TOC, suspended solids, oil-grease concentrations of MW membrane effluent were 6400 mg/L, 2592 mg/L, 320 mg/L and 270 mg/L, respectively [24], but using UF

membranes on the treatment of OMWs, its possible to obtain excellent COD removal efficiency about 96% [25] as well as obtain a clear permeate rich in polyphenols.

Recently, the recovery and fractionation of different phenolic classes from winery sludge using UF was developed by Galanakis et al. [26], the winery sludge was generated from red grapes, the study used three membrane types (1, 20 and 100 kDa), specially 20 kDa membrane retained high percentages (60%) of polar solutes (phenolic and sugars), whereas the one of 100 kDa allowed their separation from pectin and hydrolyzed derivate and the final membrane 1 kDa (fluoropolymer material) shows more selectivity, it separated successfully hydroxycinnamic acid derivate from anthocyanins and flavonols in the diluted and concentrated extract, respectively. The high selectivity of the 1 kDa membrane is due to its narrow membrane pores, in case of 2-1 kDa membranes are part of ultrafiltration to the border of nanofiltration [18].

The UF was used for concentrating polyphenols from grape seeds [27], prior to the membrane treatment, the polyphenols from the seeds was extracted using a solution with 50% ethanol and 50% water, two membranes with pore size 0.22 and 0.45 μm were used, finally the extraction of polyphenols with 0.2 g/ml solid/liquid ratio, the membrane with pore size of 0.22 μm obtained the maximum amounts of polyphenols (11.4 % of the total seeds weight), in this study the authors concluded that with the concentration step of UF, the procedure provided high extraction rates. Castro-Muñoz & Yáñez-Fernández [28] have also recovered phenolic compounds and sugars from nixtamalization wastewaters using ultrafiltration. Concerning to sugars were recovered using 100 kDa membrane (retentate with 7.72 mg/mL) and phenolic were recovered in permeate stream (951 mg/ L) by 1 kDa membrane.

Some additives as an anionic surfactant has been used in micellar ultrafiltration treatment on OMW, using Sodium Dodecyl Sulfate salt (SDS) and PVDF membrane in order to remove and concentrated polyphenols of this effluent, the initial fluxes of OMW processing by this method using SDS were 25.7 and 44.5 L/m^2 h under TMP of 3.5 and 4.5 bar, respectively. The rejection rate of polyphenols without using any surfactant ranged from 5 to 28%, whereas, it reached 74% when SDS was used under optimum pH (at pH 2) [29]. In this application, it is clear to see that membrane process can be coupled with other technologies in order to obtain better retention of the membranes.

Other type of aqueous system has been treated by membrane operations such as whey protein, caprine whey concentrates with high protein content (74%) and low lipid content (6%) were obtained by means of clarifications by thermocalcic precipitation followed by UF (10 kDa)-diafiltration, the clarification procedure increased the proportion of caseinomacropeptide and immunoglobulin G and decreased the proportion of β-lactoglobulin in the aggregates [30]. Application of UF on recovery of protein from brewer´s spent grain was studied by Tang et al. [31], more than 92% of the protein was retained by membranes with both MWCO of 5 and 30 kDa, the protein contents in the final product were 20.09% and 15.98%, respectively, for 5 and 30 kDa membranes compared with that of 4.86% concentrated by rotary evaporation.

UF technologies can be used in order to recover antioxidant components from orange press liquor [32], in this study was used a PSU hollow fiber membrane (100kDa), the author used a response surface methodology to optimize the operating conditions on the recovery of antioxidant compound where the maximum polyphenols rejection (28.45%) and antioxidant activity (32.28 mM Trolox) at 0.2 bar, 19.85 °C and 244.64 L/h. In addition, the author optimized the permeate flux and fouling index by response surface methodology using the same membrane in the treatment of orange press liquor [33].

1.2. Nanofiltration applications on recovery of bioactive compounds from effluents.

The recovery of total sugars, phenols and favonols from olive mill wastewaters (OMW) was investigated by Galanakis et al. [22] using nanofiltration technology, the NF membrane (pore size of 120 Da) showed retention of 98%, 91% and 99%, respectively. The high retention of NF membrane is due to its high selectivity towards micromolecules [18]. Even, NF technology is the current membrane process used in the food processing area for application on recovery high added value compounds due to its characteristics [19].

As well, the membrane techniques can be used for doing fractionation of OMWs, this attribution is because the NF membranes have fine pore sizes and the selection of low molecular compounds can be carried out [18]. i.e. Cassano et al. [34] evaluated an integrated membrane system (sequence of two UF processes followed by a final NF step) in the treatment of this OMWs to produce a purified fraction enriched in low molecular weight polyphenols (86.2mg/L), a concentrated fraction of organic substances (7936 mg/L Total Organic Carbon) and a water stream (0mg/L on total phenols) which can be reused in the extractive process of olive oil. Also, membrane process (as MF and UF) can be used as pre-treatment steps to produce a permeate stream containing phenolic components (oleuropein content of 544.5 mg/L), in order to do a bioconversion of some components (oleuropein to oleuropein aglycon) from OMWs by using a biocatalytic membrane reactor [35], several studies has been demonstrated that NF membranes are able to recover larger amount chemical oxygen demand from OMWs [36].

Other effluent stream that has been treated by integrated membrane process is artichoke wastewaters from the food processing industry, Conidi et al. [10] performed a study where was used a UF step as preliminary process in order to remove suspended solids from the artichoke extract, the clarified solution was submitted to a NF step with two different spiral-wound membranes (Desal DL and NP030), both membranes showed a high rejection towards the phenolic compounds (chlorogenic acid, cynarin and apigenin-7-O-glucoside), the Desal DL membrane presented high rejection on sugar compounds (glucose and fructose) (100%) compared with NP030 membrane (4.02%). Authors proposed a conceptual membrane-based process design that follows to produce three different valuable fractions: a retentate fraction with high antioxidant activity due to the retention of phenolic compounds (for nutraceutical, cosmetical or food applications), a retentate fraction enriched in sugar compounds (interesting for food applications) and a clear permeate reusable as process water for membrane cleaning.

Other type of by-product that has been treated with membrane technologies was the whey of the cheese production which is used as animal feed or released into the wastewaters treatment process, this whey was processed by NF, the process in one stage produced the best results from the point of treatment capacity, COD removal and protein recovery. The NF produce a permeate with COD load of 2,787mg O_2/L, and the protein rejection was about 88% [37], the main components that was associated to this COD are lactose, minerals, fats and proteins.

Moreover, NF systems were used in order to fractionate aqueous extracts from distilled fermented grape pomace [20], the experimental activities used several NF membranes such as Nanomax 95 membrane (250 Da), Nanomax 50 (350 Da), DL 2540 (150-300 Da), GE 2540 (1000 Da) and Inside Céram (1000 Da), the Inside Céram membrane was the higher rejection on total solids (90 %) and total phenols (90%) than the other membranes. On the other hand, the retentates obtained in the tested membranes was total a soluble solids content (0.22 to 1.410 g/100 g), total phenolics (0.173 to 1.090 mg GAE/ mL), total sugars (0.27 to 2.09 mg/mL) and antioxidant activity (3.42 to 22.5 mmol Trolox). The retentates were processed by solvent extraction to achieve the increasing of the properties due to the extraction process. Moreover, the same author used the retentates obtained from the UF and NF streams which were submitted to adsorption processes for recovering the antioxidant compounds [38], it can see that is possible to use membrane processes as prior step to other type of processes.

On the other hand, NF process was evaluated for the separation and concentration of phenolic compounds from press liquors obtained by pigmented orange peels [39], four different spiral wound NF membranes were characterized by different nominal molecular weight cut-off (MWCO) (250, 300, 400 and 1000 Da), the obtained results indicated a reduction of the average rejection towards sugars by increasing MWCO of the selected membranes, while the rejection on anthocyanins remained higher than 89% for all NF membranes investigated, the NFPES10 membrane (1000 Da) showed the lowest average rejection on sugar compounds and high rejections on anthocyanins (89.2%) and flavonoids (70%).

Table 1. Main bioactive compounds recovered by Ultra and Nanofiltration processes.

Compound:	*From*	*Membrane process used*	*Reference*
Antioxidant components	Orange press liquor	UF	Ruby-Figueroa et al. [33], Ruby-Figueroa et al. [32].
Phenolic compounds	Pigmented orange peels	NF	Conidi et al. [39]
	Olive mill wastewaters	UF	Cassano et al. [14,34] El-Abbassi et al. [29] Akdemir & Ozer [23, 24] Yahiaoui et al. [25]
	Fermented grape pomace	UF, NF	Díaz-Reinoso et al. [20,38].
	Nixtamalization wastewaters	UF	Castro-Muñoz & Yáñez-Fernández [28]
	Winery sludge from red grapes	UF	Galanakis et al. [26]
	Grape seeds	UF	Nawaz et al. [27]
Oleuropein	Olive mill wastewaters	MF and UF	Conidi et al. [35]

Chlorogenic acid Cynarin Apigenin-7-O-glucoside	Artichoke wastewaters	NF	Conidi et al. [10]
Proteins	Brewer´s spent grain	UF	Tang et al. [31]
	Whey of cheese production	NF	Yorgun et al. [37]
Caseinomacropeptide Inminoglobulin G	Caprine whey	UF	Sanmartín et al. [30]
Pectins	Olive mill wastewaters	UF	Galanakis et al. [22]
Sugars	Artichoke wastewaters	NF	Conidi et al. [10]
	Nixtamalization wastewaters	UF	Castro-Muñoz & Yáñez-Fernández [28]

Finally, **Table 1** summarizes different bioactive compounds have been recovered by Ultra and Nanofiltration from food wastes, the main reason to submit the food aqueous systems and effluents is to recover and conserve the natural components that represent a considerable chemical oxygen demand and the same components can be used in other functional applications in food, biotechnological as well as food industries. Basically, the most recovered components by membrane technologies from food processing industry are phenolic compounds. Basically, the polyphenols are of human interest due to its biological activity as nutraceutical and antioxidant activities [40].

GENERAL REMARKS

The potential advantages on membrane technologies over conventional methods on recovery bioactive compounds from effluents derivate from food industry has been successfully demonstrated, including improved product quality, easily scaled up and lower energy consumption, but they are generally limited for the characteristics of the membranes by the fouling and lack of longer durability. Currently, the recovery bioactive compounds from wastewaters by ultra and nanofiltration can be more expensive than other type of membrane processes but it has been demonstrated that the use of membrane technologies will bring great changes in the food industry in future with the development of membrane science and technology in order to create more selective membranes for specific components, with this review was showed large functionality of the membranes for recovering specific components and concentrated them. It is clear the fundamental the properties and characteristics of the membranes (pore size, affinity, material), the operating conditions and type of food aqueous system play an important role of the separation of components by ultra and nanofiltration membranes. Finally, the recovery of macro and micromolecules can be carried out by UF and NF membranes and it will depend on the characteristic of the membrane and the molecular weight of the solute targeted.

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