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State-of-the-art Membrane Processing of Solution Rich in Phenolic Compounds

N. Hamzah & C.P. Leo*

School of Chemical Engineering, Engineering Campus, Universiti Sains Malaysia, 14300 Nibong Tebal, Pulau Pinang, Malaysia

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ABSTRACT

Polyphenols and phenolic acids extracted from plants are natural antioxidants with high market value. However, they are susceptible to thermal processes, and a significant loss throughout food and beverage processing has been widely reported. This work reviews the state-of-the-aft membrane processing of the solution rich in phenolic compounds. Novel membrane processing allows phenolic concentration and water recovery simultaneously without using hazardous chemicals and high temperatures. Comparing pressure-driven membrane filtration processes with the advanced membrane processes at the low pressure in this review allowed the proper process selection to concentration phenolic coumpounds. Pressure-driven membrane filtration processes, namely microfiltration, ultrafiltration, nanofiltration, and reverse osmosis, have been studied. Nanofiltration membranes offer high retention of polyphenols due to their matching molecular weight cut-off. Osmotic distillation, membrane distillation and forward osmosis are membrane processes operated at low pressure. Osmotic distillation and forward osmosis require drawing solutions with osmotic pressure differences to separate water from phenolic compounds. A similar separation is attained in membrane distillation by creating vapour pressure differences. Membrane distillation without drawing solution is recommended since membrane fouling can be mitigated using superhydrophobic membranes with self-cleaning properties.

Keywords: Polyphenols, wastewater, membrane, membrane distillation

1.0 INTRODUCTION

Polyphenols and phenolic acids from plants are popular ingredients in the food and beverage industry because of their natural antioxidant content. Nutraceuticals, functional foods, and other natural health products with polyphenols have been commercialized worldwide due to increasing health concerns. The recovery and concentration of polyphenols and phenolic acids are important as they have been well-recognized for their health benefits. From 2022 to 2030, the market size of polyphenols is predicted to expand at a compound annual growth rate (CAGR) of 7.4% from a market size of USD 1.6 billion in 2021 [1]. However, most extraction methods require an excessive volume of organic solvent during extraction. The subsequent reduction of solvent in the extract involves several energy-intensive unit operations, including vacuum distillation, lyophilization, and evaporation. On the other side, processing and extracting these plant-based ingredients requires a lot of water and results in a great amount of wastewater with high phenolic content. About 1.96 L of water is required to produce 1 L of beverage [2]. At the same time, 0.5 L of wastewater is

* Corresponding to: C.P. Leo (email: chcpleo@usm.my)
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generated for each 1 L of beverage produced during the manufacturing of non-alcoholic beverages, including sparkling beverages, juices and nectars, fruit drinks, and syrup drinks [3].

More importantly, wastewater containing high phenolic content affects our water resources negatively without proper treatment. Similar to other pollutants, the presence of phenolic compounds in water sources can affect the ecosystem adversely [4]. The phenolic-rich wastewater, such as olive mill wastewater, requires adequate treatment since it contains a bad odour, high organic content, and low dissolved oxygen level. The typical olive mill wastewater contains 15 to 18 wt.% of organic compounds such as polyphenols, phenols, and tannins, besides 2 wt.% of organic compounds. The inorganic compounds include potassium salt and phosphates. The total phenol content usually falls in the range of 1-8 g/L. The olive mill wastewater is conventionally disposed, biologically treated, and/or physicochemical treated. The olive mill wastewater is treated with calcium oxide before disposal at the waterproof lagoons or applied as the liquid fertilizer for the plantation of olive trees. For biological treatment, the high phenolic content is diluted to reduce the inhibition of microorganism growth [5]. Evaporation, sedimentation, filtration, and centrifugation are common physicochemical treatment methods for olive mill wastewater.

The concentration of polyphenols and phenolic acids in wastewater has gained great interest in recent years since phenolic-rich wastewater, especially olive mill wastewater, is a nutritious feed to bioreactors and animals. Most of the operations, such as centrifugation and evaporation to concentrate polyphenols and phenolic acids require a great energy amount. The treatment of wastewater from the food and beverage industry using novel membrane technology is of great interest because membrane separation offers zero chemical usage and minor thermal changes besides concentrating phenolic compounds effectively.

2.0 MEMBRANE PROCESSING OF PHENOLIC COMPOUNDS

2.1 Pressure-driven Membrane Filtration of Phenolic Compounds

Many researchers have studied the potential of ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) in the treatment of olive mill wastewater (OMW) containing polyphenols and phenolic acids. The potential of membrane technology in the recovery of phenolic compounds has been widely studied, but most researchers also realized that membrane fouling appeared to be the major problem of filtration. Similar to other pollutants, the presence of phenolic compounds in different water sources causes membrane fouling which reduces the separation performance and economics of the fouled membrane [6]. While exploring the potential of membrane technology in phenolic compounds processing, improvement in phenolic compounds and water recovery should be considered as well. If the recovery of pure water and valuable ingredients can be simultaneously achieved, the economic potential of membrane technology in phenolic compounds processing can be further increased.

2.1.1 Microfiltration

Due to inappropriate pore size, microfiltration (MF) membranes only enabled 21% of the total polyphenols to be recovered from the winery effluents (wine lees) after aqueous extraction [7]. The membrane with a larger pore size showed a higher tendency of fouling even though the separation was less satisfactory. The recovery and fractionation of phenolic compounds from winery sludge were more successful using UF membranes with a low molecular weight cut-off (MWCO) [8]. Polysulfone membranes with MWCO of 20 kDa retained more than 60% of the phenolic compounds and sugars, but the membrane resistance increased due to the adsorption of polar solutes. Polyethersulfone (PES) membrane with MWCO of 5 kDa retained more phenolic compounds in the extract of Eucalyptus glovulus bark, which is higher than the retention of polyamide membrane with MWCO of 1 kDa [9]. Donnan's exclusion was expected to contribute to such observation. Although the surface chemistry of the PES membrane could be the major factor in this high retention, the PES membrane was also confirmed to be more susceptible to fouling than the polyamide membrane [9].

2.1.2 Ultrafiltration

UF membranes are more suitable for the separation of protein from polyphenol and anthocyanin extracted from purple sweet potato, as reported by Zhu *et al*. [10]. The polyphenol content in the permeate was increased from 83.4% up to 99.6%. However, the PES membrane was mainly fouled by intermediate blocking. The separation of protein from kiwifruit juice was also reported to be more encouraging using cellulose acetate membrane with MWCO of 30 kDa compared to the retention of phenolic compounds, which is only 13.5% [11].

2.1.3 Nanofiltration

NF membranes were rated to be the most appropriate candidate for the recovery of polyphenol compounds based on size exclusion. For instance, NF90 and NF270 membranes were frequently reported in the literature for the concentration of propolis extract [12], winery effluents [13], pequi aqueous extract [14] (, and olive mill wastewater [15]. More than 90% of the polyphenol compounds could be recovered using these membranes at high pressure, but fouling by lipids, protein and carbohydrates was inevitable. NF with tangential flow configuration was even optimized to concentrate the total phenolic content in olive-oil-washing wastewater to 1315.7 mg/L at 26.5 bar, 35 °C and pH 3.7 [16]. Sanches *et al*. [17] conducted a techno-economic evaluation of a NF pilot plant for olive mill wastewater. The concentration of phenolic compounds was estimated to cost up to USD$ 3.35 for every 1 m^3 of wastewater.

2.1.4 Reverse Osmosis

The RO membrane, RO98pHt removed 95.5% of COD and 94% of phenols from paper mill effluent in UF/RO configuration [18]. The integration of UF, NF and RO membranes could concentrate the total phenolic content in olive mill solid waste-pomace near 225 mg/L [19]. Flux decline was observed due to the adsorption of phenolic and hydrocarbon at an oil content of 771 ppm [20]. Helt crystallization was proposed to solidify the phenolic compounds selectively at specific freezing points after RO and vacuum evaporation [21]. The crystallization was not affected by the presence of glucose.

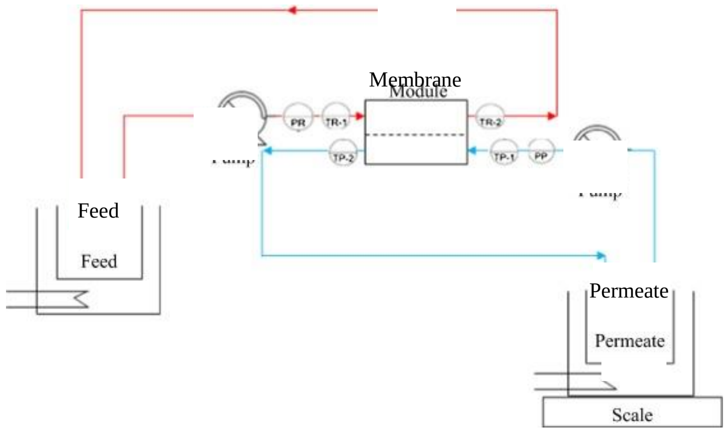

Figure 1 OD, MD, and FO set-up for laboratory studies [22]

2.2 Membrane Processing of the Phenolic Compounds at low Pressure

Due to the high-pressure requirement in NF, osmotic distillation (OD) and membrane distillation (MD) were further explored to achieve energy and cost savings in the recovery of polyphenols. A microporous membrane with great hydrophobicity works as the barrier for the feed solution, allowing the water in the vapour phase to pass through the membrane pores under the driving force of a partial vapour difference into the hypertonic solution (OD) or cold solution (MD), as shown in Figure 1. Similarly, forward osmosis (FO) also removes water from a phenolic compounds using an osmotic pressure gradient, similar to OD. However, FO consists of a semi-permeable membrane instead of a porous membrane.

2.2.1 Membrane and Osmotic Distillation

Microporous PTFE and PVDF membranes were widely applie in OD and MD. Polyphenol content in apple or beet juice was well retained during juice dehydration in OD at room temperature [23]. The organic residue on membranes caused a drop in surface hydrophobicity and porosity. The permeation flux also decreased due to membrane fouling and wetting. On the other hand, MD was successfully applied in the recovery of phenolic compounds from table olive mill wastewater [6], [24] and table olive wastewater [25]. An excellent separation factor which is higher than 95% was achieved, but severe fouling was also observed for some membranes at a high temperature. Among the commercial PTFE membranes, Kiai *et al.* [25] commented that the membrane with the smallest pore size possesses more resistance to irreversible fouling. PVDF membranes with large pore size and great hydrophobicity offer high permeate flux, but fouling by pore plugging could affect the mass transfer in the long run.

In the production of tomato paste, MD and OD retained better colour value and ascorbic acid compared to evaporation [26]. MD had also been successfully applied in the concentration of aloe vera juice and black currant juice. The flux of 14–18 kg/m2h was achieved in the concentration of aloe vera, but the flux decline over time was observed at a high concentration [27]. The black currant juice with an initial content of

22° Brix could be concentrated up to 58.2° Brix using MD [28]. An integrated membrane system with MD was predicted to further reduce cost in the concentration process of black currant juice by as much as 43% [29]. MD is actually less popular in the concentration of food and beverages compared to OD. Alves and Coelhoso [30] commented that MD produced a lower permeate flux and retained less aroma compounds in orange juice at an elevated temperature compared to OD. However, Khayet and co-workers [31], [32], [33], recovered the phenolic compounds successfully from olive mill wastewater using MD. The direct contact membrane distillation (DCMD) operated at a temperature as high as 60 °C showed insignificant effects on the phenolic content and antioxidant activity of retentate [31]. In the later study, they observed more decline of permeate flux using PTFE membrane with a bigger pore size in OMD [32]. However, the membrane fouling had not been studied in detail.

Similarly to other applications in MD, the concentration of food and beverages or MD process with high phenolic content is also affected by membrane wetting and fouling. When MD was applied to treat the high phenolic content of table olive mill wastewater [31], [32], [33], membrane fouling was reported as well. PVDF membranes with large pore size and great hydrophobicity offered high permeate flux, but pore plugging could diminish the permeate flux in the long run. Commercial PTFE membrane with a smaller pore size was reported to be more resistant to irreversible fouling [25]. Frequent cleaning of membranes could help to recover the permeate flux of a MD system used to concentrate orange juice [34]. In the coupled system of OD and MD, pomegranate juice [35] and red grape juice [23] were even concentrated by heating the feed solution and cooling the hypertonic solution simultaneously. Although permeate flux of this coupled system was higher than osmotic distillation, a similar flux decline pattern was observed due to membrane fouling and wetting (Onsekizoglu, 2013). Superhydrophobic membranes are recommended to minimize fouling in membrane distillation during the concentration of phenolic-rich solution, as shown in Figure 2 [36]. Photocatalyst could be further embedded into the membranes for cleaning purposes [37]. Hence, MD was further studied for the concentration of phenolic-rich solution such as orange juice [34], broccoli juice [38], apple juice [39], and olive mill wastewater [40, 41].

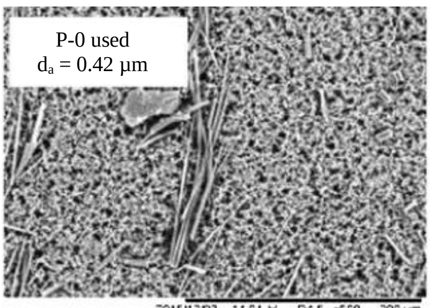

Figure 2 Membrane fouling during the distillation of gallic acid solution [22]

Table 1 Comparing membrane processes for the recovery of phenolic compounds

Membrane process	Advantages	Disadvantages
Microfiltration	High permeate flux	Low recovery
Ultrafiltration	High permeate flux	Low recovery
Nanofiltration	High recovery	Low permeate flux
Reverse osmosis	High recovery	Low permeate flux
Osmotic distillation	Low pressure	Requires drawing solution, and nonwetting membrane
Membrane distillation	Low pressure	Requires nonwetting membrane
Forward osmosis	Low pressure and minimum fouling	Requires drawing solution

2.2.2 Forward Osmosis

In FO processes, different types of drawing solution have been used to separate phenolic compounds. Singh *et al*. [42] used $MgCl_2$ solution and thin film composite FO membranes containing aquaporin to remove water from distillery wastewater. This draw solution was also selected in the alcohol removal from kiwi wine using FO [43]. Phenolic content increased to 91.59 µg/mL. About 90% of melanoidin was retained with water removal up to 70% was attained. In the concentration of grape juice, FO integrated with evaporation could increase the juice concentration from to 65.7°Brix [44]. The integration eliminated phenolic degradation due to evaporation. Fermentation of table olive processing was utilized as the draw solution in FO after UF to achieve dilution during the treatment of anaerobic disgestor sludge [45]. The COD content was reduced nearly half during UF. About 89% of organic matter and 85% of phenolic compounds were removed from this diluted stream in NF.

Compared to other membrane processes, the FO process is preferable in the recovery of phenolic compounds as long as there is proper regeneration of the drawing solution (Table 1). However, nonwetting membranes such as superhydrophobic membranes can reduce fouling in membrane distillation. The fast development of nonwetting membranes is expected to promote the use of membrane distillation for recovering phenolic compounds in the near future.

3.0 CONCLUSIONS

Membrane processing of phenolic-rich solution had been extensively developed using different types of membrane unit operations. The early works involved pressure-driven MF, UF, NF and RO. NF with high rejection of phenolic compounds and satisfactory permeate flux was highly recommended, but fouling could not be avoided under pressure. Novel membrane processes such as OD, MD and FO were explored to separate phenolic compounds at low pressure. Membrane distillation could concentrate the solution rich in phenolic compounds and recover water without using drawing solution, unlike OD and FO. However, the non-wet membrane with a superhydrophobic surface and self-cleaning properties was recommended to minimize the wetting and fouling by phenolic compounds. Membrane fouling in membrane distillation could be promoted by large pores. Hence, the permeate flux in membrane distillation could be improved using a membrane with high porosity instead of large pore size.

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