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Latest development in low-pressure osmotic-based membrane separation for liquid food concentration: a review

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Low-pressure osmotic-based membrane separation, such as osmotic membrane distillation (OMD) and forward osmosis (FO), is a separation process suitable for the concentration of liquid food with heat-sensitive components. In FO, a dense hydrophilic membrane is used with osmotic-pressure gradient as the driving force. Reverse salt diffusion and low osmotic gradient are major phenomena impeding the FO application. Food preservatives can be applied as the draw solutions, however, it is imperative to ensure the limit of food additives for safe consumption of the concentrated product. In OMD, a porous hydrophobic membrane is employed, and vapor-pressure difference acts as the driving force. Wetting is the major drawback for OMD industrialization that needs to be resolved. The application of dense membrane in OMD for liquid food concentration to overcome the wetting is highlighted in this study.

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Introduction

Liquid food concentration, such as fruit juice and other liquid foods, aims to reduce packing, storage, and transportation costs while preserving food. Liquid food concentration is usually conducted by using thermal-based processes. However, the elevated operation temperatures might lead to losing nutritional properties, especially for heat-sensitive compounds [1]. Therefore, the food industry is increasingly looking for nonthermal alternative concentration technologies. Reverse osmosis (RO) is considered an exciting alternative in liquid food processing [2–4] and offers several attractive advantages [5]. However, RO operation is limited by the osmotic pressure. Thus, the final product concentration is still less than those obtained by the evaporation method [6].

Another promising method for liquid food concentration is the low-pressure osmotic-based membrane technology, which covers forward osmosis (FO) and osmotic membrane distillation (OMD). In FO, a dense hydrophilic membrane is used as the selective barrier, and a draw solution (DS) with a higher solute concentration than the feed is employed to extract water from the feed solution (FS). FO can produce high product concentration at low operating pressure, but it is challenged by reverse salt diffusion, which contaminates the product, organic fouling, and internal concentration polarization (ICP) [7,8]. OMD, which combines membrane distillation (MD) and the osmotic process, is another alternative. In OMD, the water vapor in the FS passes through a hydrophobic porous membrane [9]. High final product concentration with preserved nutritional properties is achievable [10], thus leading to numerous research on OMD applications. The major challenge in OMD operation is concentration polarization, temperature polarization, fouling, and pore wetting, which lead to poor separation and concentration performance.

Reviews on low-pressure osmotic-based membrane separation, such as the fundamentals [11,12], fouling and control [13], and agro-food applications [14••], have been thoroughly discussed. A significant increase in publications on juice and food concentration within the last decade could be attributed to the growing consumer demand for minimally processed liquid food. Therefore, it is crucial to discuss the recent application and provide a future perspective on the development in this area, as covered in this review. The application of modified membrane, including the potential of dense OMD application to overcome the wetting issue, is highlighted. Dense MD/OMD has gained much interest in desalination application but has not been much explored for liquid food application. In the final part, future outlooks of FO and OMD in liquid food concentration are pointed out.

Food-concentration technologies: state of the art

Recent techniques to concentrate liquid foods can be classified into four different processes, that is, (i) by application of heat; (ii) by removal of heat; (iii) by the usage of gas hydrate; and (iv) by application of the membrane. The heat application is usually conducted by energy-intensive evaporation that can produce products with relatively high concentrations [15••]. Microwave and ohmic heating are two recent heat sources used to replace steam [16–19], though the applications are still limited on laboratory scale. Food concentration by removing heat, such as freeze-concentration technique, allows the removal of water from food solution by freezing the water and removing the formed high-purity ice crystals. This technique requires low temperatures, hence, it is suitable for heat-sensitive food [20], and proved to be able to preserve 90% of vitamin C and aromatic substances with no loss in color and reducing sugar during apple juice concentration [21]. Other than apple juice, pineapple juice [22], sucrose, and maltodextrin solutions [23] were also successfully concentrated by using this technique. However, other than heat-sensitive food, freeze-concentration applications are still limited due to their high capital investment [24].

Another technique that has been recently developed is CO_2 gas hydrate. This technique employs CO_2 gas molecules to stabilize water molecules at high pressure and low temperature. The small voids of gas hydrates can trap water molecules and separate water from concentrated food. Hence, sensitive constituents can be kept unchanged [25,26]. Up to now, tomato, orange, and apple juices have been produced using CO_2 gas hydrate technique [27–29]. Despite its potential, hydrate technology requires further studies to reduce processing time and increase the growth rate of gas hydrates [29]. In the last decade, membrane separation for liquid food concentration has gained much interest [7]. Several membrane processes have been attempted to produce food concentrates, such as nanofiltration [30], RO [31], FO [32,33], MD [34,35], OMD [36], pervaporation, and the combination of membrane processes [31]. Both laboratory and industrial scales of membrane-based processes for food concentration have been implemented. Excellent product quality and nutrient preservation are reported, particularly on those produced by low-pressure osmotic-based membrane operation. However, the widespread application of membranes in food concentration needs to be enhanced by tackling specific challenges during operation [7].

Principles of low-pressure osmotic-based membrane separation

FO and OMD are widely studied for liquid food concentration. FO is a separation process based on osmotic pressure gradient as the driving force for mass transfer through a hydrophilic semipermeable membrane. High-concentration DS and FS are circulated in the membrane module, hence resulting in the concentration gradient as the driving force for water transport from the feed to the DS (Figure 1(a)). The main challenges in FO operation are (i) concentration polarization, both internally and externally, (ii) reverse solute diffusion (RSD), and (iii) fouling. The discussion on FO application for liquid food concentration has been reviewed in [7].

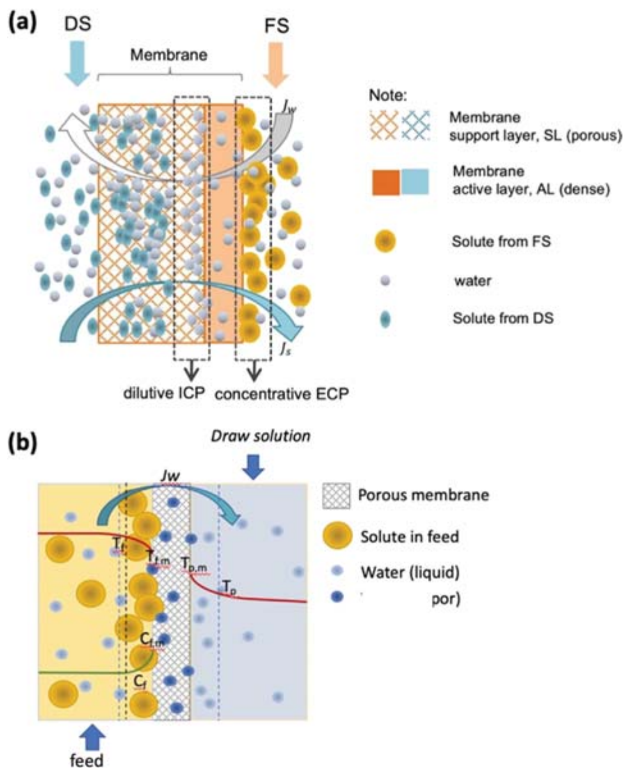
OMD is a further development of MD. In MD, porous hydrophobic membrane is used and acted as the interface separating the FS and the permeate line. The FS is heated (as low as 30°C), the permeate line is set at specific conditions that results in lower vapor pressure than the FS [37] generating vapor-pressure gradient as the driving force. Owing to membrane hydrophobicity, only volatile compounds can pass through the membrane pores and complete solute rejection is achievable [38]. The vapor-pressure gradient can also be generated by circulating a highly concentrated DS with high osmotic pressure in the permeate line. This operation is referred to as OMD (Figure 1(b)) [39]. OMD can be conducted at ambient feed temperature, thus suitable for liquid food concentration, in particular for feed that contains heat-sensitive components [40]. The challenges of MD and OMD operation are (i) concentration polarization, (ii) temperature polarization, (iii) fouling, and (iv) pore wetting. The fundamental of MD and OMD has been extensively discussed in [41]. Both FO and OMD share many benefits for liquid food concentration, such as operation at low temperature and can be operated with high feed concentration. Nevertheless, OMD does not suffer from salt back diffusion, obviating product contamination.

Application of low-pressure osmotic-based membrane separation for liquid food concentration

Forward osmosis

FO was applied in the concentration of lycopene in watermelon juice. A 4.14-fold increase in concentration

Figure 1



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Schematic illustration of **(a)** water transport in FO (ECP: external concentration polarization, Jw: flux (Reproduced with permission from 7) and **(b)** vapor transport in OMD (T_f: feed temperature in bulk solution, $T_{f,m}$ = feed temperature in feed-membrane interface, T_p: DS temperature in permeate-membrane interface, $T_{p,m}$: DS temperature in bulk solution, C_p: solute concentration in bulk FS, $C_{f,m}$: solute concentration in feed-membrane interface).

by 70–80% water removal was reported using hydrophilized polyamide membrane (Figure 2(a)) [42]. FO has also been employed to concentrate the natural pigment of beetroot to up to 12-fold. Compared with the juice concentrated by thermal evaporation, higher stability of betalains in FO-concentrated juice was reported [43•]. Concentration of dairy lactose, skim milk, and whey is also a point of interest on FO application [44,45]. A pilot-scale plant with 12-m^2 cellulose triacetate (CTA) membrane was operated to concentrate the skim milk and whey, resulting in final solid concentration of 21% and 15%, respectively. The specific energy required for this operation was in the range of 5–10 kWh/t water removed, which was lower than that required by RO [45].

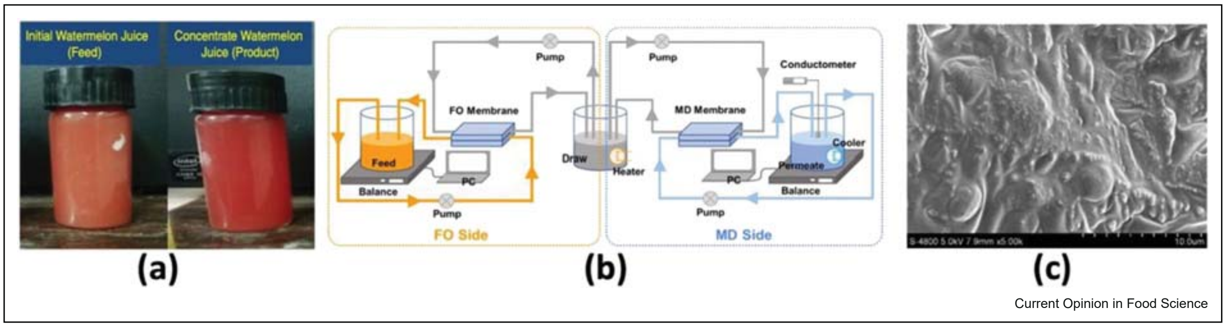
ICP, external concentration polarization, and fouling are the drawbacks that compensate the FO flux. The membrane module's vibration to enhance the flux was investigated, and flux enhancement of up to 70% was obtained [48]. Severe fouling due to the pectin has also been reported in apple juice concentration using commercial CTA membrane (Figure 2(c)). Various cleaning methods were assessed, and the results indicated effective fouling removal by a simple flush of deionized (DI) water [47]. In another study for orange juice concentration, fouling prevention can be done by microfiltration [49].

The main challenge in FO for liquid food concentration is the RSD as it may contaminate the final concentrated products. The flux of RSD is affected by the membrane being used. Higher DS flow rate and counter-current direction were reported to reduce the salt back diffusion in the test using aquaporin membrane [50]. At optimized FO operation conditions, the concentration of sugarcane from 150 g/L to 531 g/L was conducted with an energy consumption of 92.14 W/L of water removed [51]. The lack of a suitable DS with good stability, low price, nontoxicity, and minimal reverse salt permeation has led to the application of food preservatives as the DS [46]. However, the limit of the food preservative concentration in the concentrated product should be maintained below its regulatory limit to ensure safe consumption. Regardless of the draw solutes being used, their concentration increased over time and reduced the driving force for mass transfer. In a recent study, FO was coupled with MD (Figure 2(b)), the latter serves to reconcentrate the DS, enabling continuous concentration of apple juice in long-term operation [46]. In another study combining FO and evaporation, FO acted as the preconcentration step. The retention of bioactive compounds of the combined processes was greater than the stand-alone evaporation process, highlighting the potential of FO as a preconcentration step [15••].

Membrane distillation and osmotic membrane distillation

Membrane distillation (MD) and osmotic MD (OMD) have been studied for the concentration of fruit juice, sugarcane, and dairy products, however, recent studies in MD and OMD have led toward the concentration of fruit juice. The RO and MD networks have been opted for as an alternative to multi-effect evaporation to minimize fouling and energy costs in milk concentration. The study indicated RO utilization until the milk concentration reached 18 wt%, followed by single-stage air gap MD (AGMD). However, the operation faced several challenges, such as fouling, the high-energy requirement for heating and cooling, and energy cost to achieve sufficient cross-flow velocity (Moejes et al., 2020). Vacuum MD (VMD) has been employed to concentrate sugarcane juice to obtain sugar crystals. Using 0.012-m^2 polypropylene (PP) membrane, 50 mL of sugarcane model solution with initial concentration of 49°Brix was concentrated to 73°Brix in 15 h of operation, and sugar crystals are visible (Figure 3(a)) [52]. MD has also been utilized for fruit juice concentration with feed

Figure 2



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(a) Images of the initial and concentrated watermelon juice (Reproduced with permission from [42]), **(b)** schematic setup of FO–MD configuration (Reproduced with permission from [46]), **(c)** SEM image of pectin-fouled membrane surface during apple juice concentration (Reproduced with permission from [47]).

temperatures ranging from 30 to 50°C (Figure 3(b)). The trade-off between nutrient content and permeate flux was highlighted, which indicated the importance of operation at low feed temperature [34]. This leads to the application of OMD for fruit juice concentration [34,53]. The juice concentration, DS concentration, and Reynolds number affect the driving force for mass transfer, while the membrane pore size affects the mass transfer coefficient in OMD operation [53].

Regarding the energy consumption required to recover the permeates in fruit juice concentration, a comparison between the VMD and OMD was conducted at similar juice-concentration factors. The energy consumption per produced permeate in OMD operation was 60% higher than that in VMD operation (4893 J/g in OMD compared with 3090 J/g in VMD) as it involved two-stage phase changes (Figure 3(c) and (d)). Therefore, strategies to recover the diluted DS are vital for the industrial application of OMD for fruit juice concentration [55]. OMD has also been applied in the production of high-protein- concentrated whey beverages. The targeted soluble solid content of 15.7°Brix could be achieved from initial concentration of 5°Brix in 240 min of OMD using 0.22 GVHP polyvinylidene fluoride hydrophobic membrane (Millipore, Ireland), with retained nutritive value and acceptable clarity [56•]. Concentration of pomegranate juice using polyvinylidene fluoride (PVDF) and polytetrafluoroethylene (PTFE) OMD was also reported high-quality concentrated juice, as indicated by the excellent values of phenolic content, flavonoid content, and antioxidant activity [57].

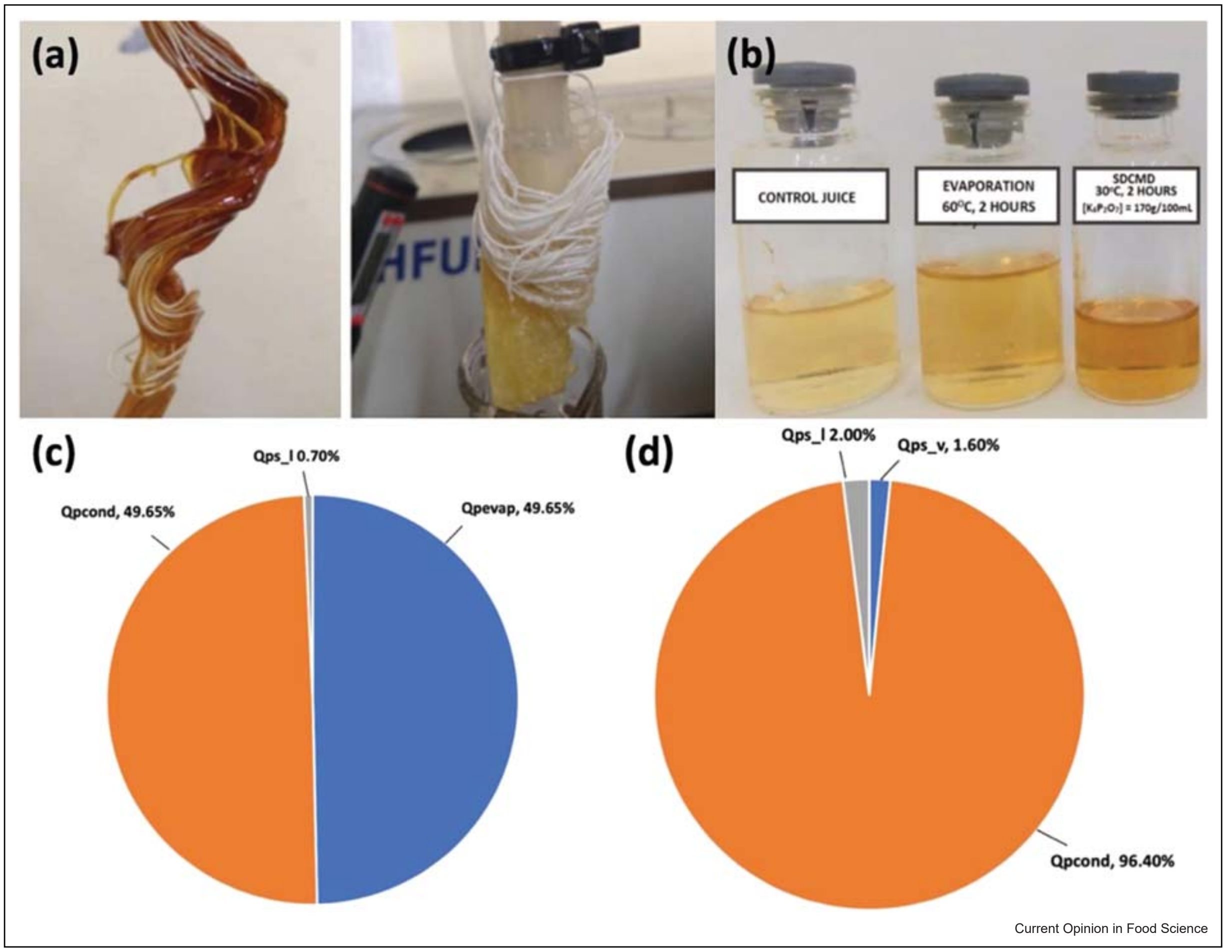
One of the crucial challenges in OMD for liquid food concentration is the low-permeate flux. While it can be compensated by increasing the membrane surface area [34], many studies operated the OMD at a prolonged duration to achieve the required final concentration, which can be detrimental to the nutrient quality. In a recent study, the OMD to concentrate Nagpur mandarin was coupled with ultrafiltration (UF) and RO, which served as the clarification and preconcentration stages. The ascorbic acid and antioxidant in the juice were retained, suggesting the configuration as the alternative to the currently employed thermal evaporation process [58••]. Plasma-modified RO membrane with higher flux than the commercial thin-film composite membrane was used, resulting in 30% reduction of OMD operation time to obtain 60°Brix pomegranate juice [59]. The combination of OMD and MD was also examined to concentrate bioactive anthocyanins from muscadine grapes. To enhance the driving force and permeate flux, high-concentration brine was used as the DS. At the same time, the feed temperature was also elevated to 40°C. The anthocyanins can be concentrated up to three-fold, however, the adsorption of anthocyanins on the membrane surface posed another challenge that needs to be resolved for optimized operation [60].

Most studies in MD and OMD for liquid food concentration were conducted using commercially available membranes, which are PP, PTFE, and PVDF. It is worth noting that severe fouling occurs in OMD for liquid food concentration, which leads to the pores blocking and reduced permeate flux [36]. Fouling could simultaneously reduce the permeate flux by more than 20%, 50%, and 70% during the concentration of apple juice, sugarcane solution, and whey [34,52,56•]. Wetting was also reported in the sugarcane juice and pomegranate juice concentration, impeding the separation process [36,52]. Detailed data of studies in MD and OMD for liquid food concentration are presented in Table 1.

Latest development of membrane modification in osmotic membrane distillation

Modifying membrane structure and material to alleviate fouling and wetting has been conducted in other MD

Figure 3



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(a) Sugar crystal in concentration of sugarcane juice using MD (Reproduced with permission from [54•]), **(b)** visual observation of concentrated apple juice at various concentration method (Reproduced with permission from [34]), **(c)** energy-consumption contributor in OMD at 10 mbar, and **(d)** energy contributor in VMD at 10 mbar (Q_{pcond} = energy consumption for permeate condensation, Q_{pevap} = energy consumption for permeate evaporation in DS, Q_{ps_l} = sensible heat in heating/cooling of the liquid permeate, Q_{ps_v} = sensible heat in cooling the vapor permeate).
Figure 2c and d are reproduced with permission from [55].

and OMD applications. In the study of MD and OMD for other applications, specifically tailored membranes, such as superhydrophobic, omniphobic, and Janus membranes, have been widely utilized [62–64]. However, in the application of OMD for liquid food concentration, utilization of membranes other than those commercially available is still lacking. Recently, a dual-layer hierarchical fibrous composite (HFC) membrane fabricated via electrospinning process was used in pomegranate juice concentration. The thin active layer and thick support layer consisted of poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) and poly(ethylene terephthalate) (PET), respectively. Even though gel-layer formation was observed in the concentration of pomegranate juice, the fouling layer was easily removed by DI water. The flux reduction of the operation was also not as severe as the commercial PVDF and PTFE membrane. Interestingly, the results showed significant improvement in the OMD flux using the modified membrane, which indicated an increase in mass transfer through the modified membrane [61••].

Excellent fouling preventions were reported in many studies, yet, pore wetting still occurred, particularly in long-term experiments. To prevent wetting, the utilization of dense membrane for MD has been studied and thoroughly discussed [65]. To date, most of the studies on the application of dense MD and OMD were conducted for desalination purposes. However, dense OMD, specifically those with composite structure, possess great potential to be applied in liquid food concentration. Composite dense OMD consists of porous hydrophobic support layer and a dense hydrophilic top layer. The dense top layer allows liquid water to

Table 1

Selected studies in liquid food concentration by low-pressure osmotic-based membrane separation.

Membrane technology	Feed	Membrane material and structure	Flux	Concentrate quality	Concentration factor	Ref
FO	Watermelon juice	Polyamide-polyether sulfone	Initial flux of 13.5 LMH, reduced to 13 LMH in 10 h.	96.01% lycopene recovery	4	[42]
FO	Beetroot juice	CTA flat-sheet membrane	Initial flux of 12 LMH, reduced to 1.29 LMH in 12 h.	High betalain concentration. Betalain degradation was 3 times slower than the thermal evaporation method.	12	[4-3•]
FO	Sweet whey	CTA and aquaporin flat- sheet membrane	Initial flux of 20 LMH, significantly reduced after reaching total solute concentration of 18%.	Preserved nutritional quality	> 4	[44]
FO	Whey	CTA	Initial flux of approximately 4 LMH	Insignificant reduction of protein, fat, and lactose concentration after FO.	2–2.5, depends on the initial composition of the whey.	[45]
MD	Apple juice	PP hollow fiber	Initial = 0.73 LMH, 20% flux reduction in 2 h	3% and 5% loss of vitamin C and phenolic compounds, respectively.	≈3	[34]
MD	Sugarcane juice	PP hollow fiber	Initial = 0.2 LMH, flux reduced to 0.05 LMH after 10 h of operation, before wetting.	Final concentration of 63°Brix.	1.3	[52]
OMD	Pomegranate juice	PVDF and PTFE flat sheet	PVDF: Initial flux of 1.246 LMH, final flux of 0.208 LMH. PTFE: Initial flux of 2.077 LMH, final flux of 0.935 LMH	Preserved nutritional quality	PVDF: 1.8 PTFE: 3.9	
OMD	Whey (from cheese making)	Durapore 0.22 GVHP polyvinylidene fluoride hydrophobic membrane	Initial = 14 LMH, 57% flux reduction in 320 min	1.5-fold increase in essential amino acid concentration.	≈2.7	[5-6•]
OMD	Pomegranate juice	The thin active layer and thick support layer consisted of poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) and poly (ethylene terephthalate) (PET). Liquid entry pressure = 25 kPa. Contact angle = 143.7°.	Initial = 8.621 LMH at 25°C (4-times higher-than- reported commercial membranes). Flux reduced to 0.312 LMH after 25.5 h of operation.	Final product concentration of 51.3°Brix	3.6	[6-
1••]						

permeate through the solution-diffusion mechanism, followed by water-phase change in the interface of the top and support layer. The water vapor then passes through the hydrophobic membrane pores, resulting in pure water separation from the FS with no risk of pore wetting. Dense OMD was fabricated with polyvinyl alcohol as the top layer and commercially available porous layer. To model the fruit juice concentration, a FS containing sucrose and limonene oil was used at various concentrations. Good membrane stability and superior antiwetting properties were reported [66]. Despite its potential, the study of dense OMD for liquid food concentration is limited and more research should be conducted toward this particular topic.

Future outlook

Membrane separation techniques have been attempted to concentrate liquid food. As they are operated under mild conditions, preservation of nutritional compounds is ensured. Low-pressure osmotic-based membrane separation, such as OMD and FO, has been extensively studied. OMD is a membrane-based process that exploits a porous hydrophobic membrane and vapor-pressure gradient to extract water in the form of vapor from the liquid food, while FO employs a dense hydrophilic membrane and osmotic gradient to draw liquid water from the liquid food.

The availability of suitable DSs is vital for FO operation. DSs with characteristics of nontoxic, low salt back diffusion, and high osmotic pressure, are required. Recently, food additives were utilized as novel DSs. Though salt back diffusion still occurred, the novel DS is safe to consume. However, it is imperative to ensure the limit of food additives for safe consumption of the concentrated product. Low permeate flux is still a major concern in FO, thus, the utilization of a modified membrane in the application of liquid food concentration is needed.

Salt back diffusion is absent in OMD, ensuring the purity of the product. However, Low permeate flux, membrane fouling, and wetting challenged OMD application and should be addressed for its industrial application. Removal of foulant by pretreatment step may be an alternative as a fouling control approach. The development of modified membranes, such as omniphobic and Janus membranes, has been devoted to solving those membrane issues. Furthermore, the dense membrane has also gained much interest as its structure prohibits wetting. However, the study of dense OMD for liquid food concentration is very limited, despite its great potential.

DS regeneration is vital to maintain OMD and FO performance. The hybrid process, such as OMD-Evaporation and FO–MD, has been employed for continuous liquid food and DS concentration to achieve stable flux. However, the setup requires high-energy consumption for DS heating and cooling. Alternative strategies for DS reconcentration are important in realizing the economically feasible liquid food concentration by osmotic-based membrane separation.

CRediT authorship contribution statement

Helen Julian: Writing – original draft; **K. Khoiruddin**: Writing – original draft; **Putu D. Sutrisna**: Writing – original draft; **Siti Machmudah**: Writing – original draft; **I G. Wenten**: Conceptualization, Writing – original draft.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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- • of special interest
- •• of outstanding interest.

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This study investigated the potential of FO as pre-concentration step during grape juice concentration. FO was combined with conventional evaporation, and this combined process was compared to a stand-alone evaporation. This study shows that FO can concentrate grape juice to 65.7°Brix. The combined FO-evaporation process exhibits higher retained bioactive components in the concentrated juice than the stand-alone evaporation

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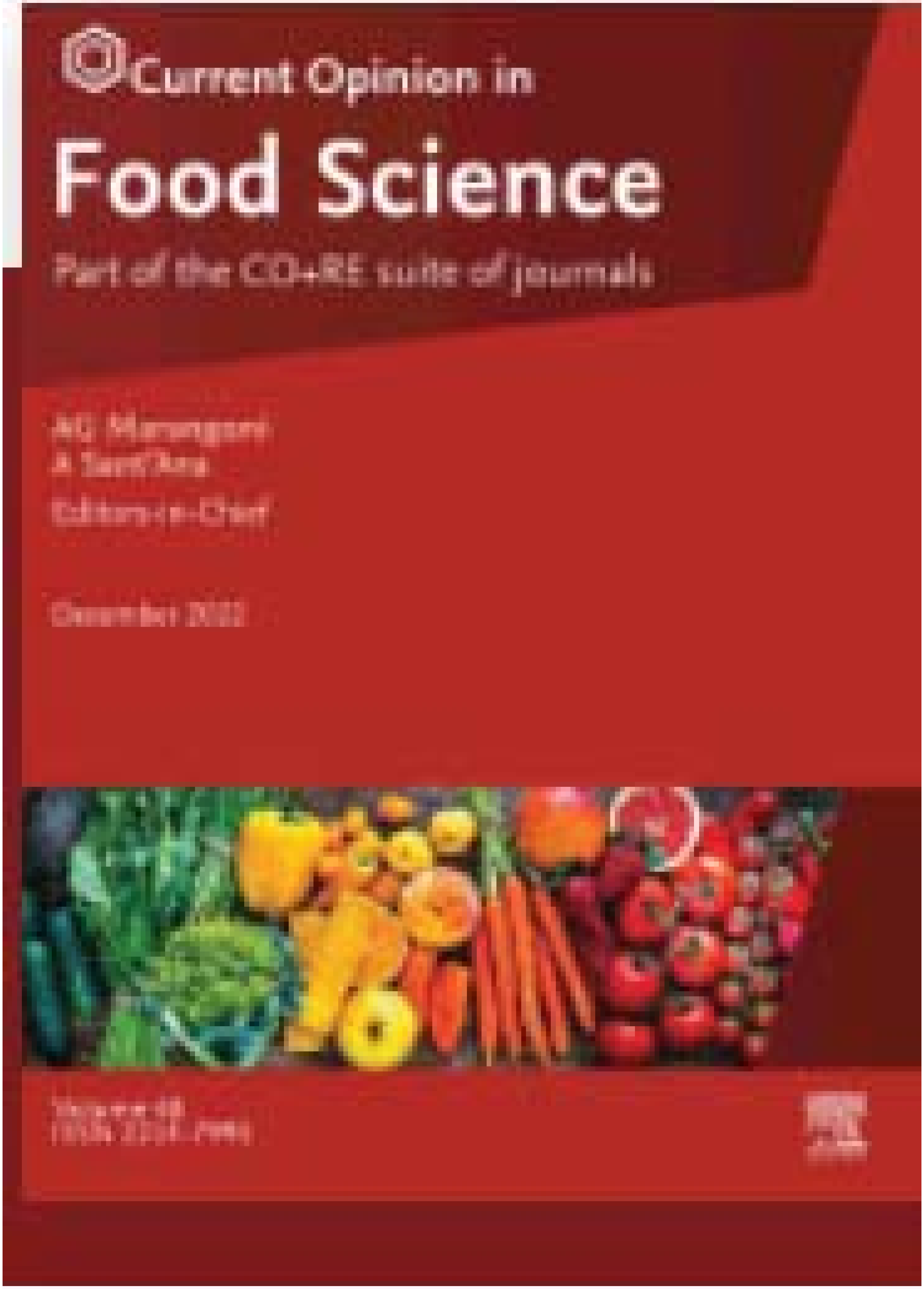

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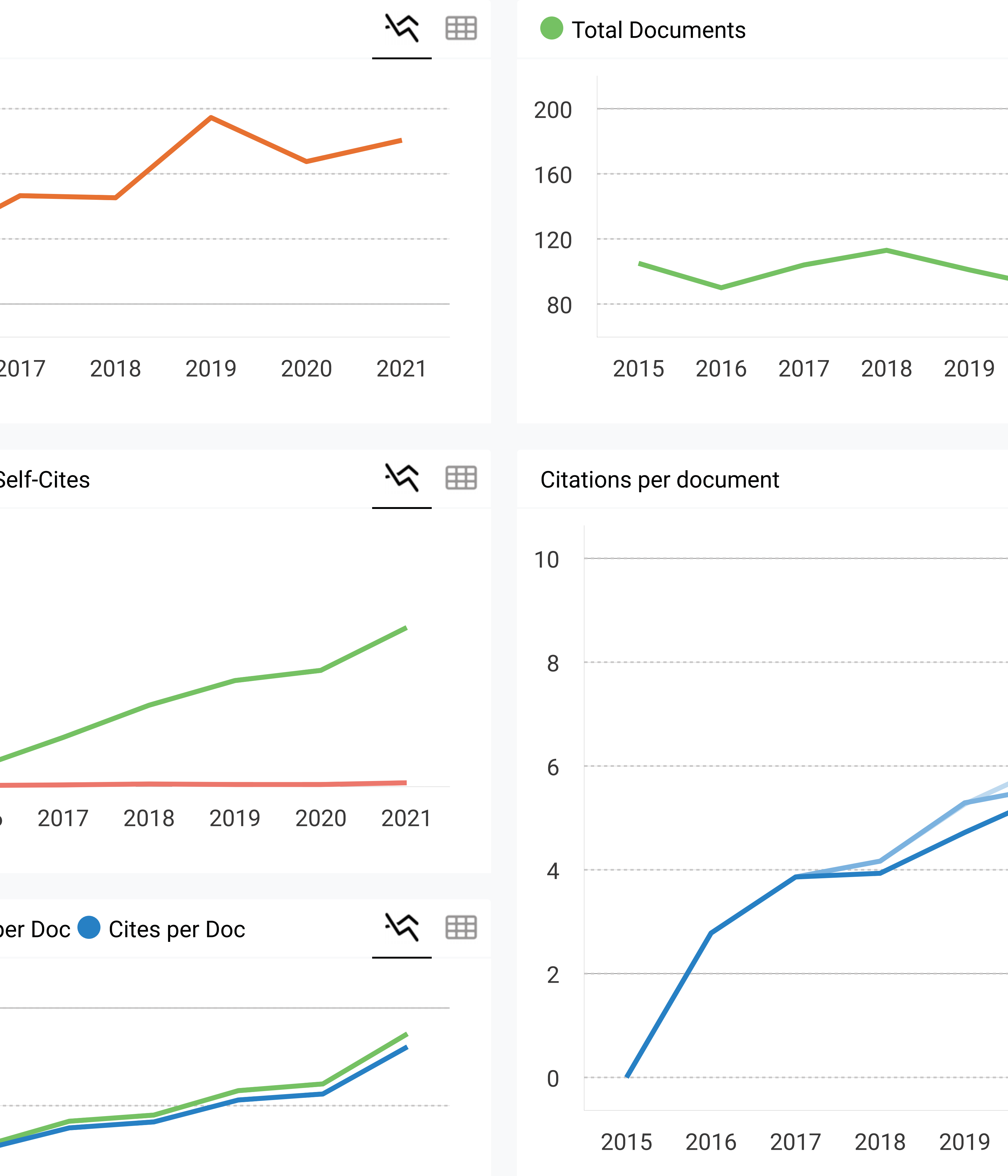
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