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MEMBRANE TECHNOLOGIES IN THE OIL INDUSTRY AND THEIR POTENTIAL APPLICATION FOR THE RECOVERY OF PHYTONUTRIENTS FROM PALM OIL

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ABSTRACT

Palm oil is one of the most widely commercialised vegetable oils globally. This oil contains important phytonutrients, such as carotenoids (provitamin A), tocopherols and tocotrienols (vitamin E). These phytonutrients are thermally degraded and removed from crude palm oil (CPO) through bleaching techniques in the refinement process. This article focuses on using membrane technology as a green alternative to recover these compounds, specifically, the use of nanofiltration as a potential mechanism for recovering edible oil phytonutrients from palm oil. Furthermore, due to the few studies related to this topic, the review also highlights microfiltration and ultrafiltration uses in other crude oils (CO) for degumming and deacidification refining processes for phospholipids (Pl) and free fatty acids (FFA) removal. Finally, an overview of the biological functions of palm oil phytonutrients in health and their applications in the food industry is presented.

Keywords: microfiltration, nanofiltration, palm oil, phytonutrients, ultrafiltration.

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INTRODUCTION

Over the years, the food industry has faced new challenges, mainly population growth. According to the United Nations, the world's population is expected to increase by approximately 2.0 billion people to 9.7 billion people by 2050 (United Nations, 2019). This has forced the food industry to research and generate high nutritional content for populations (Cárdenas, 2016). Ways of making this possible include making agro-industrial processes sustainable, better utilising various techniques and recovering nutritional compounds from generated residues and byproducts thereby reducing the environmental impacts of processing while obtaining value-added compounds (Almaraz-Sánchez *et al.*, 2022).

The palm oil industry is one of the most important agro-industrial chains leading to commercial vegetable oil (Cárdenas, 2016; Statista, 2021). From this chain, palm oil is the main product that is obtained from the mesocarp of the fruit via extraction by mechanical pressing. Crude palm oil (CPO) has a characteristic red-orange colour due to compounds that are rarely

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present in other foods (Sylvester and Shah, 2004) such as carotenoids, tocopherols, and tocotrienols (Dong *et al.*, 2017). Each compound has nutritional and bioactive functions and must be part of a balanced diet. However, traditional refinement processes (Mosquera *et al.*, 2009) thermally degrade carotenoids and remove tocopherols and tocotrienols (Amado, 2010).

An alternative method for recovering these compounds is by using membrane technologies to separate solutes according to the molecular size by adjusting the pressure conditions, temperature and characteristics of the membrane such as the molecular weight cut-off (MWCO), material and molecule-membrane interactions (Mohammad *et al.*, 2015). This method has multiple advantages over conventional separation techniques such as molecular distillation, supercritical fluid extraction or adsorption, because it does not require the intensive expenditure of energy, conserves selected compounds in the oil (Azmi *et al.*, 2015; Bernardo and Drioli, 2010), is scalable and has a compact design (Kang and Cao, 2014; Mohammad *et al.*, 2019; Otitoju *et al.*, 2016).

Membrane technologies have various applications in the food industry, for example, in the manufacturing processes of dairy products for milk protein recovery (Chamberland *et al.*, 2019; Deshwal *et al.*, 2020; Gavazzi-April *et al.*, 2018; Lauzin *et al.*, 2020; Li and Corredig, 2020; Lobasenko *et al.*, 2019; Renhe and Corredig, 2018; Rodrigues Toledo Renhe *et al.*, 2019; Sánchez-Moya *et al.*, 2020; Schäfer *et al.*, 2019; Shakhno *et al.*, 2019; Sousa *et al.*, 2019; Touhami *et al.*, 2020; Uttamrao *et al.*, 2019; Valencia *et al.*, 2018), the clarification of beverages (Akhtar *et al.*, 2020; Bindes *et al.*, 2020; Destani *et al.*, 2020; Rodrigues *et al.*, 2020), and the recovery of enzymes and metabolites of biotechnological processes (Balti *et al.*, 2018; Díaz-Montes and Castro-Muñoz, 2019; Díaz-Montes *et al.*, 2020; Ter Beek *et al.*, 2019; Todero *et al.*, 2019; Wang *et al.*, 2018; Zhang *et al.*, 2016).

Since 1990, the application of this technological data to the oil industry resulted in a higher academic publication rate in some years than in others. However, there has been an increase in publication rates associated with this topic. According to the Scopus database, these publications have been oriented toward improving the oil refinement processes by membrane technologies using microfiltration, ultrafiltration and nanofiltration for antioxidant separation (Mohammad *et al.*, 2015), degumming (phospholipid removal) (Ariono *et al.*, 2018), deacidification (Azmi *et al.*, 2015; Lai *et al.*, 2016; Purwasasmita *et al.*, 2015), the removal of traces of metals, products of oxidation, and sterols (Iyuke *et al.*, 2004) and decolourisation in relation to carotenoid separation (Ghazali *et al.*, 2022; Manjula and Subramanian, 2007; Reddy *et al.*, 2001). In these articles, there is a particular interest in studying flux, rejection, fouling mechanisms, solute-membrane interactions, membrane material, operative conditions, and feed characteristics due to the high viscosity of oils. This represents a challenge for the use of membrane technology in this agribusiness.

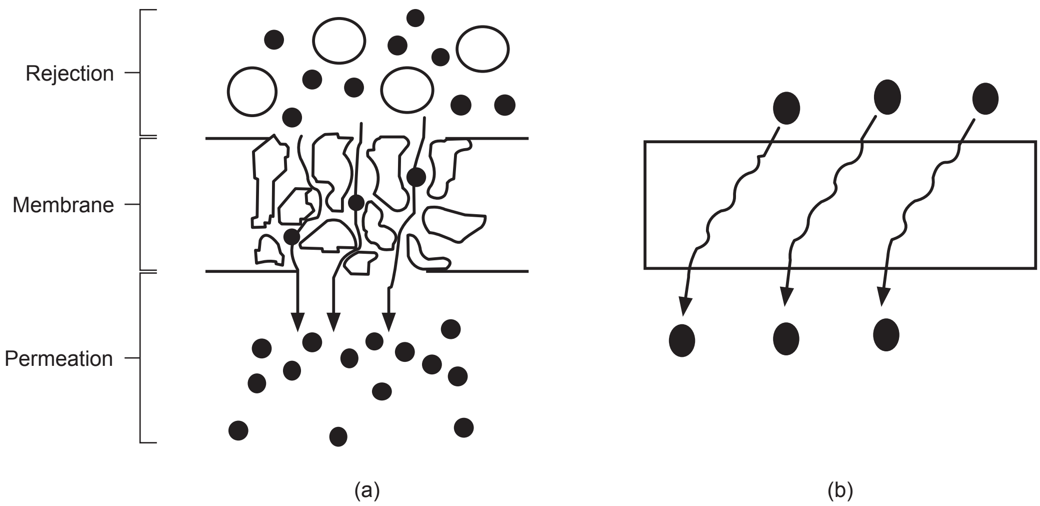
To the best of our knowledge, there are few reviews about applying membrane technologies in CPO for minor components recovery, such as the one published by de Morais Coutinho *et al.* (2009). Chiu *et al.* (2009) and Darnoko and Cheryan (2006a) focused on using membranes for carotenoid concentration. There were few publications from 2009 until 2021 when this topic gained relevant attention again (Ghazali *et al.*, 2022; Lim and Ghazali, 2021; Nabu *et al.*, 2021). For this reason, it is necessary to carry out a new overview of the studies conducted in recent years on membrane technology for the oil refinement process and the recovery and concentration of phytonutrients (specifically from palm oil), which could have applications in the food industry.

The first part of the review corresponds to the separation process principles of membrane technology. Recent applications of this technology in the edible oil industry are then reviewed. Finally, the prospects for palm oil phytonutrients recovered by nanofiltration in functional food formulations are proposed.

MEMBRANE TECHNOLOGY: SEPARATION PROCESS PRINCIPLES

The membrane filtration process involves the application of a driving force on a feed flow with high solute concentration. This driving force enables the separation of molecules or components according to the membrane MWCO and specific pressure ranges: Microfiltration separates particles in the range 0.025-10.000 μm at pressures <0.2 MPa; ultrafiltration separates particles in the range 1000-300 000 Da at pressures >1 MPa; and nanofiltration separates particles in the range 350-1000 Da at pressures of 1-4 MPa (Baker, 2012b; de Morais Coutinho *et al.*, 2009; Solís *et al.*, 2017).

These separation processes are conducted through the membrane by pore flow and solution-diffusion models (Baker, 2012a). According to this, microfiltration and ultrafiltration permeants are transported by the pore flow model, while permeation in nanofiltration can be explained by both models in *Figure 1* (Baker, 2012a).



Source: Baker (2012a).

Figure 1. (a) Pore-flow model and (b) solution-diffusion model.

Within the parameters in the separation process, the retention coefficient or rejection (R) is an important parameter to consider. It is expressed as the rejection rate of the solute in terms of the concentration of the solute in the permeate (C_p) and the concentration of the solute in the retentate (C_R) [Equation (1)]. The flux (J) is expressed as the ratio of the volumetric flow (F_p) of the permeate that is obtained during the process to the area (A) as a function of time [Equation (2)] (Baker, 2004; 2012a; Belfort *et al.*, 1994; Chew *et al.*, 2020; Doran, 2013; de Morais Coutinho *et al.*, 2009).

$$R = (C_R - C_P)/C_R = 1 - C_p/C_R \quad (1)$$

$$J = F_P/A \quad (2)$$

The flux can also be determined using Darcy's law for cases in which cake formation has occurred due to a high concentration of solutes on the membrane surface; such a scenario offers additional resistance to the flux:

$$J = (1/A)(dV/dt) = \Delta\rho/\eta_o\,(R_m+R_c) \quad (3)$$

where (J) denotes the flux, V is the total volume of the permeate, A is the external area of the membrane, t is the filtration time, $\Delta\rho$ is the pressure drop that is imposed through the membrane and cake, (R_m) is the membrane resistance, and (R_c) is the resistance due to cake formation (Doran, 2013).

The concentration factor (F_c) is another critical parameter and it is the ratio of the mass in the feed flow (M_A) at the beginning of the process and the mass in the retentate (M_R) at the end of the process [Equation (4)]. The membrane permeability (JV) is the ratio of the volume of the permeate (V) to the permeation area of the membrane (A), multiplied by the filtration time (t) and the operating pressure (P) [Equation (5)] (Doran, 2013).

$$F_C = M_A/M_R \quad (4)$$

$$JV = V/A\,t\,p \quad (5)$$

The transmembrane pressure ($\Delta\rho$) is the main driving force that directs the filtration process through the membrane. It corresponds to the differential pressure between the feed flow pressure (P_F) and the permeation pressure (P_P) [Equation (6)] (Doran, 2013). However, the driving force can be affected by the osmotic pressure of solutes present in the feed [Equation (7)]. The π_F and π_p values correspond to the feed osmotic pressure and permeation osmotic pressure, respectively. For this case, both can be calculated by the Van´t Hoff equation, where C is the molar concentration of the solute, R is the ideal gas constant, T is the absolute temperature, and i is the number of ions that form as the molecule dissociates [Equation (8)] (Chew *et al.*, 2020; Doran, 2013).

$$\Delta p = p_F - p_P \quad (6)$$

$$\Delta\, p = (p_F - p_p) - (\pi_F - \pi_p) \quad (7)$$

$$\pi = iCRT \quad (8)$$

Regardless of which membrane technique is employed, challenges are encountered during the filtration process, which can be summarised as: Gel layer formation, fouling, pore plugging, adsorption, and concentration polarisation. The latter occurs when there is an accumulation of particles in the feed near the membrane, which can be estimated by the parameter Ψ [Equation (9)], where C_0 corresponds to the concentration of solutes in the feed stream, C_w is this concentration after the dead-end filtration and R is the retention coefficient. In this case, the flux is expressed by Equation (10), where C_p corresponds to the solute concentration in the permeate and k is the mass transfer constant in cm s^{-1} (Chew *et al.*, 2020).

$$\Psi = C_w/C_0 - 1 = 1/(1 - R) \quad (9)$$

$$J = k\ ln\ (C_w - C_p)/(C_0 - C_p) \quad (10)$$

In contrast, fouling during the filtration process occurs due to the interaction between solutes that are suspended in the feed flow and the membrane, where factors such as the size of the suspended solutes, hydrophobicity, isoelectric point, Brownian motion, van der Waals forces, electrostatic charge, and Lewis acid-base interactions affect the process (Mohammad *et al.*, 2015; Pérez and Labanda, 2013). In addition, other characteristics of the operating system such as the flow regime (laminar or turbulent), affect its formation (Kilduff *et al.*, 2002); these characteristics also include the feed flow and type of filtration (dead-end or crossflow). In addition, to describe the fouling behaviour during filtration, the selectivity of the membrane, the capacity (which refers to the permeation flux per unit area and the phenomenon of mass transfer), and the momentum in the process must be considered (Chew *et al.*, 2020). In general, fouling can be developed through four mechanisms: Cake formation, pore constriction, intermediate blocking (n=1) and complete blocking (n=2) (*Figure 2*) (Chew *et al.*, 2020; Wenten *et al.*, 2019). For each case, the Hermia model can be applied, which describes the behaviour of the permeate flux in relation to time, as expressed in Equation (11), where denotes the filtration time, V the volume of the permeate, and the index or fouling type, which can be selected according to the pore blocking category (*Figure 2*) (Aryanti *et al.*, 2017; Chew *et al.*, 2020; Wenten *et al.*, 2019).

$$d^2t/dV^2 = k\ (dt/dV)^n \quad (11)$$

For each case, a distinct model has been established, according to Ariono *et al.* (2018):

Complete blocking (n=2) $\quad lnJ = lnJ_0 - k_c t \quad$ (12)

Intermediate blocking (n=1) $\quad 1/J = 1/J_0 + k_i t \quad$ (13)

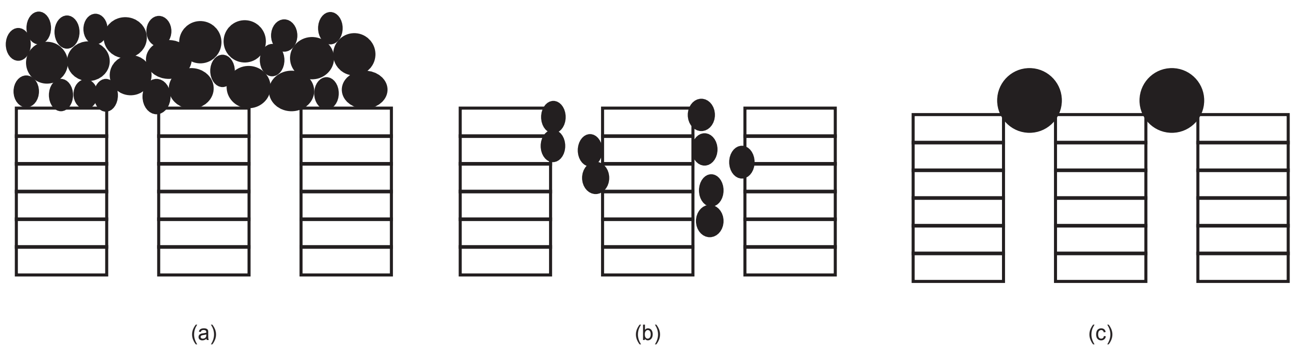
Pore constriction (n=1.5) $\quad 1/\sqrt{J} = 1/\sqrt{J_0} + k_s t \quad$ (14)

Cake formation (n=0) $\quad 1/J^2 = 1/J_0^2 + k_{cf} t \quad$ (15)

where k_c, k_i, k_s, and k_{cf} denote the respective constants for each case of membrane fouling.

In addition to these models, Chew *et al.* (2020) revisited the research by Kilduff *et al.* (2002), and summarised classic models of flux behaviour according to the type of fouling and classic models when cross- or dead-end filtration was used. In addition, new models have been proposed for cases in which classical models do not fully explain the fouling behaviour.

To describe a real flux behaviour, the resistance in the series model considers the viscosity (μ), pressure drop (ΔP), resistance offered by the



Source: Wenten *et al.* (2019); Aryanti *et al.* (2017) and Chew *et al.* (2020).

Figure 2. (a) Cake formation (n=0), (b) pore constriction (n=1,5) and (c) intermediate blocking (n=1) - complete blocking (n=2).

membrane (R_m), the resistance provided by fouling (R_f), and additional resistance due to concentration polarisation (R_c) [Equation (16)]. However, other aspects must be considered that are not included in the model, such as experimentation with the techniques of characterisation of the cake in real time to understand the molecular mechanism that occurs during the filtration process and its dependence on time (Chew *et al.*, 2020).

$$J = \Delta P/\mu(R_m + R_f + R_c) \qquad (16)$$

As an alternative to the resistance in the series model, the concentration polarisation can be measured in terms of mass transfer theory or the hydrodynamic effect of the boundary layer, as devised in film theory (Ghazali *et al.*, 2022). However, by maintaining turbulent flow conditions, the solute concentration can be kept constant (C_B). When this does not occur, the highest concentration of solutes is found close to the membrane wall (C_w). Additionally, during this process, the thickness of the concentration in the boundary layer, δ, which depends on the concentration gradient of these solutes to and from the membrane, must be considered (Ariono *et al.*, 2018; Chew *et al.*, 2020; Othman *et al.*, 2021). If a static state is considered, this behaviour is expressed by Equation (17), where the rate of transport of solutes on the surface of the membrane is related to the rate of transport of solutes to the permeate minus the diffusivity rate in the higher concentration of solutes in the boundary layer, according to Fick's law (Chew *et al.*, 2020; Doran, 2013).

$$JC - JC_P - Ɗ\,(dC/dy) = 0 \qquad (17)$$

In this equation, C is the concentration in the boundary layer, C_p is the concentration in the permeate, Ɗ is the diffusion coefficient, and dC/dy is the solute concentration gradient with respect to the distance (y). Integrating this expression yields Equation (18), where Ɗ/δ corresponds to the mass transfer coefficient, which is expressed as Equation (19). When the process pressure rises, the concentration in the membrane wall (C_w) could reach a limit value C_G that corresponds to the formation of a polarisation gel on the surface of the membrane, and this corresponds to the expression in Equation (20) (Chew *et al.*, 2020; Doran, 2013).

$$J = Ɗ/\delta\ ln\ (C_w - C_p)/(C_B - C_p) \qquad (18)$$

$$J = k\ ln\ (C_w - C_p)/(C_B - C_p) \qquad (19)$$

$$J = k\ ln\ (C_G/C_B) \qquad (20)$$

RECENT APPLICATIONS OF MEMBRANE TECHNOLOGY IN THE EDIBLE OIL INDUSTRY

Microfiltration

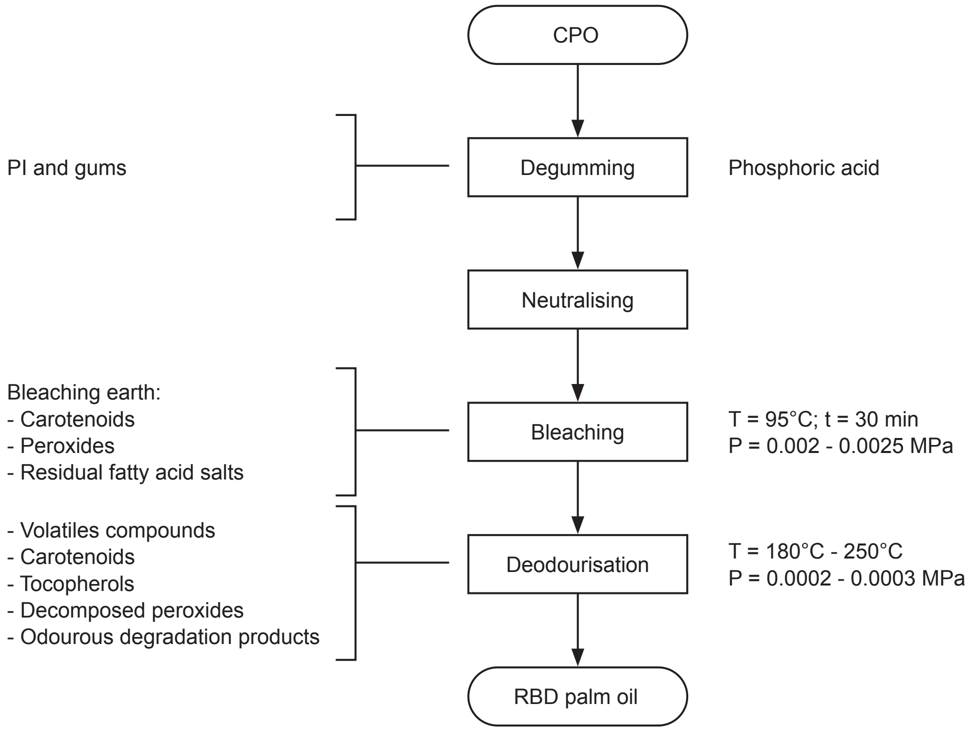
Degumming. Studies of the microfiltration process for Pl removal in CPO are few, although other edible oils, have been studied (Bassam *et al.*, 2001; 2004; 2009; 2011; Hafidi *et al.*, 2005; Hou *et al.*, 2020). Edible oil refinement involves degumming, neutralisation/deacidification, bleaching, and deodourisation to remove FFA, Pl, pigments and other compounds that affect the physicochemical quality, appearance, taste, functionality, and shelf life (*Figure 3*) (Doshi and Shah, 2018).

The degumming process is one of the first steps in refining edible oils and consists of removing Pl and waxes from CO (Corley and Tinker, 2016). However, Pl can form emulsions with the water from clarification, and separation becomes more complicated, resulting in deterioration and low oil quality (Doshi and Shah, 2018).

Likewise, in soybean oil extraction, organic solvents such as hexane increase the probability of reverse micelle formation due to the surfactant nature of Pl (Hou *et al.*, 2020). These micelles can reach MW of 20 000 Da or more (Boynueğri *et al.*, 2017), which simplifies the separation process by membrane technology.

Under traditional refining conditions, these micelles are removed by centrifugation (Hou *et al.*, 2020) but due to the high energy consumption of this process, membrane technologies have become an alternative method for removing these molecules (Doshi and Shah, 2018; Hou *et al.*, 2020). Microfiltration is an excellent option because it performs solid-liquid separation (Baker, 2012a). The membrane performance can vary according to the transmembrane pressure, flow rate, feed temperature, membrane MWCO, and solid concentration in the feed stream (Bernardo and Drioli, 2017). Under these conditions, microfiltration removes large molecules such as phosphatides that are traditionally removed during oil refinement. According to databases, the application of membrane technologies for degumming processes has been maintained over time and is considered an effective method for these processes. Another microfiltration application is FFA and peroxide removal (Hou *et al.*, 2020).

Membrane materials that are used in the degumming process. In the late 1990s, there was a demand to use membrane processes in this phase. As a result, patents were generated, such as those reported by Bassam *et al.* (2001), who proposed a polyacrylonitrile (PAN) polymer membrane with a 0.3 µm MWCO for removing Pl from soybean oil micelles. In 2004, 2009, and 2011, the same authors



Note: RBD - refined, bleached and deodourised oil.

Source: Corley and Tinker (2016).

Figure 3. Traditional palm oil refining process.

patented the use of PAN microfiltration membranes to separate and purify compounds as soy lecithin products as a result of this process (Bassam *et al.*, 2004; 2009; 2011). In recent years, the use of synthetic membranes has increased, and these include polyethersulfone (PES) (Ariono *et al.*, 2018), polyamide (PA), polysulfone (PS), polyvinylidene fluoride (PVDF), polyimide (PI), PAN, and zirconium dioxide-ZnO_2 (ceramic). However, these membranes have primarily been used in microfiltration processes (Hou *et al.*, 2020; Mestre *et al.*, 2019; Roy *et al.*, 2014). Synthetic membranes outperform organic membranes because difficulties are encountered with organic membranes under operating conditions, especially the prolonged use of organic solvents, which reduces the stability and, therefore, mechanical resistance (Hou *et al.*, 2020).

Ceramic materials have shown various advantages because of their resistance to high pressures and temperatures. By comparing the performances of membranes according to their capacity for removing Pl from oils using microfiltration and ultrafiltration, ceramic membranes present a higher flux during filtration than other materials, which is close to 50 L h^{-1} m^{-2} at 0.25 MPa, under similar operating conditions (Hou *et al.*, 2020). In the case of ultrafiltration for degumming processes, this value is higher than those registered in PVDF (MWCO: 6000 Da) and PES (MWCO: 22 000 Da) (Ochoa *et al.*, 2001), thereby demonstrating the advantages of using membranes with larger pore sizes and ceramic materials for this purpose.

Membrane performance - Fouling. Membrane fouling can decline due to the influence of the membrane MWCO. This behaviour can be represented by four precise models (*Figure 1*) in which membranes with smaller pore sizes could lead to a decline in flux in a shorter time than membranes with a higher MWCO, as Hou *et al.* (2020) determined in their research. Hou *et al.* (2020) studied Pl removal from crude rapeseed oil. In this study, the cake model realised the best fit for a hydrophilic ceramic membrane of 0.05 μ MWCO, followed by the intermediate model. In contrast, the intermediate model realised the best fit for the same sample using a ceramic membrane of 0.20 μ MWCO, followed by the cake model. It was also found that a combination of these two models better fit the explained fouling behaviour. Hence, the phenomenon can be explained through these two mechanisms. This finding also demonstrates the incidence of MWCO on membrane fouling

TABLE 1. APPLICATION OF MICROFILTRATION MEMBRANES IN PHOSPHOLIPID REMOVAL FROM DIFFERENT EDIBLE OILS

Crude oil	Phospholipid initial content (mg kg^{-1} oil)	Membrane material	MWCO (µm)	Filtration	T (°C)	Transmembrane pressure (MPa)	Phospholipids rejection (%)	Phospholipid final content (mg kg^{-1} oil)	Authors
Rapeseed	5 851.75	Ceramic membrane: ZrO_2 Support material: α-Al_2O_3	0.2	Crossflow	55	0.15	99.9	1.4	Hou *et al.* (2020)
Rapeseed	5 851.70	Ceramic membrane: ZrO_2 Support material: α-Al_2O_3	0.2	Crossflow	42	0.10	99.9	1.3	
Soybean	545.00	PAN	0.3	-	-	1.00	96.1	21.0	Bassam *et al.* (2011)
Corn oil	1 783.00	PAN	0.3	-	-	1.00	97.6	41.2	
Canola oil	505.00	PAN	0.3	-	-	1.00	95.1	24.4	
Sunflower previous neutralised 20% NaOH	43.00	Cellulose	2.5	Dead end	25	0.20	98.1	-	Hafidi *et al.* (2005)
Sunflower previous neutralised 40% NaOH	43.00	Cellulose	2.5	Dead end	25	0.20	83.7	-	

and Pl micelle rejection. In addition, the authors suggested that it is also affected by the increase in transmembrane pressure, which causes the pores to block and consequently, decrease the flux. Another interesting finding from this research was the increase in fouling with temperature because temperature affects the micelle diameter. However, a temperature increase promotes inverse diffusion of the micelles from the membrane surface to the oil, thereby decreasing the mass transfer coefficient.

Deacidification. Specifically in CPO, Azmi *et al.* (2015) evaluated polyvinyl alcohol (PVA) and PVDF microfiltration membranes for FFA removal. In this research, the hydrophobic membrane significantly facilitates the removal process due to its characteristics, while PVA membranes with low hydrophobicities limit the phospholipid separation process. Thus, materials such as PVDF (with higher hydrophobicity) could have a valuable role in the process (Azmi *et al.*, 2015).

However, the application of microfiltration is not limited to the removal of FFA from oil. Over the years, advances in this topic have been extensive, and new membrane materials have been developed. This opens the possibility for further exploration. The affinity between the compounds to be removed or preserved and the membrane is an essential factor to be considered. Azmi *et al.* (2015) also evaluated the performance of PVA crosslinked PVDF for FFA removal from CPO due to this affinity. FFA corresponds to a derived carboxyl that shows affinity and selectivity for hydroxyl groups. PVA is a material that has abundant hydroxyl groups; hence, it is expected to have a high concentration of FFA in rejection. However, in this research, a high concentration of PVA on the surface of the membrane had adverse effects on the fatty acid removal process since a high fixation of the material could block the surface, thereby suggesting that having just a thin layer of this material on the membrane surface probably has the desired effect on FFA removal.

Other applications of microfiltration membranes are in the separation of oil/water emulsions to remove only the oil phase to purify the water; in this case, the membrane surface can be modified by introducing hydrophilicity. Materials such as PS can be modified with a layer of polydopamine. Membranes with this modification show better separation performance than membranes that have not been modified (Bernardo and Drioli, 2017).

Ultrafiltration

Ultrafiltration enables the separation of colloids and microparticles with particle sizes close to 1000-300 000 Da (Baker, 2012b; Solís *et al.*, 2017). Similar to microfiltration membranes, these membranes have been applied in the oil industry for processes such as deacidification (Azmi *et al*., 2015; Gonçalves *et al*., 2016), degumming (Ariono *et al*., 2018; Aryanti *et al*., 2017) and solvent removal from micelles (Saravanan *et al*., 2006).

Ultrafiltration has been used in the edible oils industry to remove Pl and FFA, and the complex effects of these compounds on fouling formation have been studied (Aryanti *et al*., 2017). However, micelles also impact fouling during the ultrafiltration process. According to Ariono *et al.* (2018), micelles have an affinity for other polar components, even impurities, which bind and contribute to the formation of fouling. In addition, because Pl molecules are amphiphilic, they can be absorbed by the membrane and contribute to fouling formation. Pl are compounds that form micelles when dispersed in water (Aryanti *et al*., 2018).

Degumming. Studies in CPO have removed Pl with high rejection, above 87.3%. These results are comparable with residual phosphorus in traditional refined palm oil, not above 2 ppm (Othman *et al*., 2021). These results were achieved by adding an inorganic adsorbent in a mixed matrix PVDF membrane, where increasing magnesium silicate, from 3.0% to 8.0% in the matrix, increased the phospholipids rejection.

In addition to the materials, temperature and pressure significantly affect CPO Pl rejection. For example, PES membranes with MWCO close to 100 000 Da were tested. The results showed that as the pressure and temperature increased, the flux permeation increased while Pl were retained. The high flux permeation can be explained because the pressure deforms and enlarges the pores while temperature reduces the feed viscosity promoting the process (Razi *et al*., 2021).

Several reports have analysed the effect of material membranes for degumming CO from various vegetable lipid sources. For example, Doshi and Shah (2018), analysed modifications of PVDF hollow fibre membranes (MWCO 570 000 Da - 600 000 Da) to identify better treatment for separating micelles from crude peanut oil. In this study, the membrane composition of 15% PVDF, 75% N-methyl-2-pyrrolidinone and 10% ethylene glycol showed excellent performance with satisfactory flux (47.5 L h^{-1} m^{-2}) and 94% micelle rejection.

Boynueğri *et al.* (2017) studied the tubular PVDF membrane performance with an MWCO of 20 000 Da for degumming crude soybean oil using a reverse osmosis/ultrafiltration module. According to the results, it was possible to reject 86.11% of phospholipids, and an average flux of 17.04 L h^{-1} m^{-2} at 40°C and 2 MPa pressure was realised. To improve molecular rejection in various membrane materials, Onal-ulusoy (2015) tested PVDF and PES flat sheet membranes (MWCO 30 000 Da) that were modified with hexamethyldisiloxane HMDSO (hydrophobic monomer). This modification was conducted using radiofrequency plasma polymerisation treatment, which improved characteristics such as permeability, polarity, and selectivity of polymeric membrane surfaces, as shown by the studies conducted by Onal-ulusoy *et al.* (2014) and; Tur *et al.* (2011) in which various glow discharge powers and radiofrequencies were tested. These studies evidenced that changes in chemical and physical properties on the membrane surface were due to sequential oxidation and crosslinking reactions once the polymers were deposited in the plasma. In the same study, an exciting change was observed at large values of the contact angle (γS_w): A weak interaction occurred between the water and the membrane surface, which resulted in a hydrophobic membrane. This increase in the modified membrane surface hydrophobicity increased the percentage of rejection of each material by 6.3% and 16.8% for PES and PVDF, respectively.

Ultrafiltration membranes are also used in degumming processes in the study of colloid-enhanced ultrafiltration, or polyelectrictrolyte ultrafiltration (PEUF), and micellar-enhanced ultrafiltration (MEUF) (Danis and Aydiner, 2009; Niazmand *et al*., 2015). These processes involve using a surfactant that adsorbs undesirable components electrostatically or through the hydrophobic feed effect, thereby resulting in the formation of complex aggregates (micelles of the surfactant) that cannot pass through the membrane. Therefore, these aggregates are concentrated in the rejection stream. In this case, the surfactant consists of Pl, which is hydrated to form micelles, which facilitate the separation of these compounds from the oil (Danis and Aydiner, 2009; Niazmand *et al*., 2015).

Niazmand *et al.* (2015) studied micelle formation due to the presence of chelating agents such as ethylenediaminetetraacetic acid (EDTA) which improved the heritability of phosphorus and, in the presence of water, they observed that polyelectrolytes (with magnesium or calcium ions) can form micelles that precipitate, thereby enhancing the separation process. Niazmand *et al.* (2015) reported high efficiency in process of removing phospholipids from crude canola oil.

Deacidification. Aryanti *et al.* (2018), Azmi *et al.* (2015) and Purwasasmita *et al.* (2015) studied the application of various ultrafiltration membranes to CPO and CPO diluted with organic solvents as presented in *Table 2*, where the membrane characteristics and operating conditions are described.

On the other hand, modifications in PVDF membranes by additions of inorganic adsorbents such as magnesium silicate have been studied for

TABLE 2. USES AND MATERIALS OF ULTRAFILTRATION MEMBRANE FOR DEGUMMING, DEACIDIFICATION AND BLEACHING* PROCESSES IN CPO AND DERIVATIVES

Molecule to remove	Vegetable oil	Membrane material	Membrane characteristic	Membrane affinity	T (°C)	P (MPa)	Rejection*** (%)	Authors
Oil/water separation	CPO**	Polypropylene (PP)	Hollow fibre	Superhydrophobic	40	0.2	99	Wenten *et al.* (2019)
PI FFA	CPO**	Polyethersulfone	Flat sheet	Hydrophilic	30	0.1	99 16	Aryanti *et al.* (2018)
PI	A mixture of refining palm oil with isopropanol and lecithin (as a representation of CPO)	Polyethersulfone	-	-	24-25	0.1	79	Aryanti *et al.* (2017)
FFA	CPO**	Polyethersulfone	Hollow fibre	Hydrophilic	70	-	97	Purwasasmita *et al.* (2015)
FFA	CPO**	Polyvinyl alcohol (PVA) crosslinked poly-vinylidene (PVDF)	Hollow fibre	Hydrophilic	65	0.2	5.93	Azmi *et al.* (2015)
PI Carotenoids Tocopherols Tocotrienols	CPO** and crude palm olein	Nonporous composite polymeric membrane with silicon as active layer and PI as support	Flat sheet - - -	Hydrophobic	40	4	95-100 - - -	Arora *et al.* (2006)
Phospholipids Carotenoids Free fatty acids Volatile material	CPO**	Polyethersulfone	Tubular	-	63	2.6	96.4 15.8 0 0	Ong *et al.* (1999)

Note: *Bleaching - refining oil process where pigment is removed, mainly carotenoids; **CPO - crude palm oil; *** - The maximum rejection reported during each experiment.

FFA removal from CPO, obtaining rejections close to 16.51%, with simultaneous Pl rejection (Othman *et al.*, 2021). In this study, although FFA rejection was not as expected, there was a percentage retained due to the increasing pressure and temperature in the experiments. Similar results were obtained by Aryanti *et al.* (2018) using PES. However, in both cases, not only factors such as membrane, material and operation conditions affect the affinity and FFA rejection but MWCO also significantly affected these results.

Oil/water separation. Superhydrophobic membranes are additional membranes used for oil/water separation in CPO. The material repels water on the membrane surface due to a high-water angle (WCA) of above 150° and a low contact angle of hysteresis and a sliding angle of below 10°. This characteristic is helpful in the oil refinement process once the oil has been extracted and hot water or steam is added for hydrolysing gums and resins in CO. Traditionally, to remove this water, centrifugation is performed to clarify the oil, but an alternative is to replace this step with the use of membranes (Wenten *et al.*, 2019). In this case, a superhydrophobic membrane enables the successful removal of the water that is present in the oil.

In CPO, Wenten *et al.* (2019) used a polypropylene (PP) superhydrophilic membrane with a MWCO of 0.05 µm, to remove water from palm oil. According to the reported results, at 40°C and 0.2 MPa, it was possible to remove substantial water content and maintain a high flux and 99% rejection. The transmembrane pressure and the temperature directly impact the oil viscosity; thus, an increase in temperature decreases the oil viscosity, thereby facilitating the permeate flux. Additionally, an increase in TMP influences the flux; in this case, linear behaviour was evaluated at each temperature point.

Nanofiltration

Nanofiltration in the edible oil industry. The application of nanofiltration in the oil industry has shown a rising trend recently. In palm oil, most research is focused on water treatment after oil extraction. However, according to the Scopus database, studies related to the recovery of carotenoids or decolourisation can be an alternative option for maintaining the interest of the academic community. Despite the low number of publications, this area represents a research opportunity in this field. Although technological data were collected in the 1990s and insipient research on nanofiltration in palm oil was conducted between 2006 and 2009, few results have been published until now, with recent publications in the last three years.

Opportunity for phytonutrient recovery in CPO processing. During bleaching, in the traditional refinement process, carotenoids, chlorophylls, and other compounds, such as metals, soaps, polyaromatic compounds, Pl and a fraction of tocopherols and sterols, are removed (González-Tovar *et al.*, 2005) by using clays and bleaching earth (diatoms) at a temperature of 95°C for 30 min under vacuum conditions at 0.15-0.18 Pa (Corley and Tinker, 2016).

During this process, an adsorption mechanism occurs in the bleaching earth. Due to atmospheric factors or induced temperature and oxygen factors, the many cases of non-saturation in α- and β-carotenes produce a tendency for oxidation and isomerisation (from the E configuration to the Z configuration) (Nagarajan *et al.*, 2017). After this, the bleaching earth and these molecules are eliminated as waste, which generates an environmental impact due to aldehydes, ketones, and other compounds that are derived from the process and adsorbed by the material (Haro *et al.*, 2014). Thus, molecules with high nutritional value, such as carotenoids, tocopherols and sterols, are removed during the process. Only a small amount remains in the olein fraction (refined oil) (Loganathan *et al.*, 2020a). For this reason, nanofiltration is an alternative to the bleaching phase. This separation process consists of filtration, where hydraulic pressure is physically applied as a driving force for the transport of mass (Chiu *et al.*, 2009), in this case, palm oil is applied through a membrane. As a result of this operation, there will be a retentate stream where solutes are concentrated (Doran, 2013). In this case, the recovered compounds are carotenoids, tocopherols, and tocotrienols, which can be applied in various industries, such as food additives, functional food formulation, pharmaceutical, nutraceutical and nutricosmetic industries (Mohd-Nasir and Mohd-Setapar, 2018; Taeymans *et al.*, 2014).

Nanofiltration explicitly involves a series of hydrodynamic and interfacial microevents on the membrane surfaces and in their nanopores (Mohammad *et al.*, 2015). A combination of steric, Donnan, dielectric, and transport effects interfere with the process (Mohammad *et al.*, 2015). Solute transport is performed through a steric mechanism (size-based exclusion). According to Pérez and Labanda (2013), the nanofiltration process can also be considered 'low-pressure reverse osmosis' because it combines characteristics of reverse osmosis, where the separation mechanism is based on a solution-diffusion and ultrafiltration and operates based on pore-flow model (*Figure 1*). The interaction between the membrane and the solute charge also plays an important role.

Palm oil is a vegetable source with a high phytonutrients content (Choo, 1994; Saini *et al.*, 2015). It is estimated that palm oil can provide 15 times more retinol equivalent (provitamin A) than carrots and approximately 300 times more than tomatoes (Loganathan *et al.*, 2017; Sundram *et al.*, 2003). According to Mba *et al.* (2015) and Rincón and Martinez (2009), this content can vary between 500-700 ppm or even reach 1000 ppm for the oil of hybrid palm (Interspecific cross: *E. oleifera* x *E. guineensis* - OxG) (Chaves *et al.*, 2018), where 90% corresponds to α- and β-carotene, which are mainly in the all-*trans* state (Yaakob and Chin-Ping, 2003). These variations depend on the genotype (variety/cultivar), geography, season, agronomic management, maturation, and postharvest management conditions (Yaakob and Chin-Ping, 2003). Moreover, the content can also vary among fruits in the same bunch or bunches of the same palm (Corley and Tinker, 2016), although the synthesised retinol equivalent is simultaneously consumed in the lipid of the fruit synthesis as they mature (Prada *et al.*, 2011).

The tocopherol and tocotrienol contents are estimated to be between 600 and 1000 ppm (*Table 3*) (Chaves *et al.*, 2018; Rincón and Martinez, 2009), which corresponds to 70%-80% and consists mostly of tocotrienols (Mba *et al.*, 2015). Seppanen *et al.* (2010) and Kamal-Eldin and Budilarto (2015) used tocopherols and tocotrienols to extend the shelf life of food. These studies showed that α-tocopherols and tocotrienols generally show higher antioxidant activity than γ-tocopherols in fats and oils (Saini and Keum, 2016; Seppanen *et al.*, 2010). Studies have demonstrated that these compounds are antiatherogenic (Kirmizis and Chatzidimitriou, 2009), allergic dermatitis suppressive (Tsuduki *et al.*, 2013), hypolipidemic (Minhajuddin *et al.*, 2005), nephroprotective (Siddiqui *et al.*, 2013), anti-inflammatory (Mocchegiani *et al.*, 2014) and neuroprotective (Rashid Khan *et al.*, 2015) agents.

The molecular weights of these carotenoids, tocopherols and tocotrienols are between 396 to 536 Da. Hence, the use of nanofiltration membranes is suggested with suitable operating conditions for the recovery process (*Table 4*).

State-of-the art nanofiltration membranes that are applied in the palm oil industry. Few studies have been conducted on nanofiltration in the edible oil refinement process because this technology is considered an emerging technology that has not yet been widely exploited (Peeva *et al.*, 2010). It has been reported since 2006 and 2009 that nanofiltration membranes can be used for complete carotenoid recovery or concentration (Chiu *et al.*, 2009; Darnoko and Cheryan, 2006b). Chiu *et al.* (2009) achieved a rejection of 75% of these compounds in palm oil, which mainly included α- and β-carotene,

TABLE 3. CAROTENOID, TOCOPHEROL AND TOCOTRIENOL CONTENT IN O×G AND *E. guineensis* CPO

Compounds	Hybrid OxG (ppm)*	*E. guineensis* (ppm)**
	Chaves *et al.* (2018)	Rincón and Martinez (2009)
α-carotene	447.9-577.7	185.0-209.0
β-carotene	724.0-911.8	401.0-413.0
Total carotene	1 172.1-1449.6	586.0-622.0
δ-tocotrienol	45.6	103.0-115.0
β+ϒ-tocotrienol	666.0	640.0-665.0
α-tocotrienol	199.3	310.0-336.0
β+ϒ-tocopherol	-	-
α -tocopherol	26.8	161.0-162.0
Total tocotrienols and tocopherols	937.6	1 215.0-1 277.0

Note: * - Hybrid of two palm oil species: *E. oleifera* Coarí x *E. guineensis* La Mé; ** - *E. guineensis*, is the oil palm species with a large cultivation area. The values presented correspond to the species sampled in Western Colombia.

TABLE 4. EXAMPLES OF MOLECULES PRESENT IN PALM OIL THAT CAN SEPARATED THROUGH MEMBRANE TECHNOLOGIES

Operative pressure	Filtration	Molecular weight	Examples
< 0.2 MPa	Microfiltration	0.025 - 10 μm	Phospholipids
> 1.0 MPa	Ultrafiltration	1 000-300 000 Da	FFA
1.0 - 4.0 MPa	Nanofiltration	350-1 000 Da	Carotenoids, tocopherols, tocotrienols

Source: Baker (2012), de Morais Coutinho *et al.* (2009) and Doran (2013).

at a temperature of 40°C and a pressure of 2.5 MPa. Similar results were obtained by Darnoko and Cheryan (2006b) when testing membranes with various hydrophobicity properties. Their results demonstrated that both hydrophilic and hydrophobic membranes performed excellently in retaining carotenoid molecules. However, the hydrophobic membrane showed the best flux performance in the process, which demonstrates the value of this characteristic which is deficient in filtration process with hydrophilic membranes. The recovery of nonpolar solvents using nanofiltration membranes has also been studied, especially in oilseeds that require solvents for the oil extraction process, such as soybean. In this case, hydrophobic membranes are recommended (Darvishmanesh *et al.*, 2011).

Challenges for CPO minor components separation using nanofiltration. Another topic that is relevant and considered as a challenge for carotenoids and other minor components separation is the high viscosity of the CPO and the molecular weight of each compound. This situation has led to sample preparation with methanolic transesterification evaluated in previous studies as a good option (Chiu *et al.*, 2009; Darnoko and Cheryan, 2000). Nevertheless, in the last three years, two articles about this topic have been published. Ghazali *et al.* (2022) studied CPO diluted in different organic solvents: Acetone, ethyl acetate and n-hexane, where CPO with hexane, showed higher triglyceride rejection and low carotenoids rejections. In this case, the membrane material, MWCO and pression had an important influence in this separation. Ghazali *et al.* (2022) also determined that P84® PI membrane with 280 Da MWCO had the best performance and low pressure (1 MPa) allowed increased carotenoids rejection.

On the other hand, Lim and Ghazali (2019) analysed the combination of CPO with Organic Solvent Nanofiltration (OSN) membranes. Results indicated high triglycerides rejection as the stirring increased and at the same time, the CPO concentration in dilution with organic solvent decreased. This can be explained because the concentration polarisation that affects separation efficiency (Lim and Ghazali, 2021).

PROSPECTS FOR PALM OIL PHYTONUTRIENTS THAT ARE RECOVERED BY NANOFILTRATION TECHNOLOGY IN FUNCTIONAL FOOD FORMULATIONS

Currently, palm oil is one of the most widely commercialised vegetable oils in the world (Ariono *et al.*, 2018; Hariyadi, 2020) and palm oil and its byproducts are used as ingredients for multiple food formulations, such as frying processes, ice creams, kinds of margarine and ready-to-eat foods (Hariyadi, 2020), due to their thermal stability and functionality (Loganathan *et al.*, 2020b; Mba *et al.*, 2015).

In recent years, interest in using palm oil varieties such as red palm oil (RPO) in the food industry has been increasing, which is mainly because these compounds contain molecules with high health and nutritional values. Both α- and β-carotenes are recognised as provitamin A, with β-carotene having a stronger biological effect, and tocopherols and tocotrienols are recognised as vitamin E and have antioxidant effects. These compounds have been studied separately in intervention studies with controlled clinical trials. These studies have reported that RPO application in food formulations demonstrated a positive effect on health, namely, it increased vitamin A levels, which were measured as serum retinol concentrations, in children and maternals (Loganathan *et al.*, 2020a). RPO is produced by refining traditional palm oil via deacidification and deodourisation processes using molecular distillation to maintain 80% of the pigments and vitamins in the oil (Loganathan *et al.*, 2017).

This demonstrates the interest in examining methods for recovering phytonutrients from CPO and the potential of these phytonutrients as natural ingredients with multiple applications in the development of functional foods. In this case, nanofiltration could play an essential role because it may concentrate phytonutrients in a determinate amount of oil, even at a higher content than is typical for RPO. This opportunity gives the possibility to formulate functional food with barely perceptible changes in rheological and sensory properties.

RPO in Clinical Trials

Intervention studies have employed RPO as a vitamin A supplement. Some of these studies have demonstrated the thermal stability of this compound in high-temperature processing (180°C for 20 min), such as Loganathan *et al.* (2020a). In this research, Loganathan *et al.* (2020a) developed cupcakes with RPO and palm olein addition. The results showed that the cupcakes that were formulated with RPO and the cupcakes that were formulated with palm oil did not differ significantly in terms of stability and both had an acceptable range of rancidity (peroxide value of 7.5 mEq kg^{-1}). Additionally, there was 100% retention of phytonutrients (α- and β-carotene) in the cupcake that was formulated with RPO, in compliance with the vitamin A recommended dietary allowance (RDA).

In intervention studies, the addition of RPO yielded significant improvements in antioxidant concentrations in red blood cells and the liver (Loganathan *et al.*, 2017). In studies that were conducted by Ojeda *et al.* (2017), RPO was provided to patients during an interventional period in meals at lunch and dinner. Although the primary purpose of this study was to evaluate the functional effect of the oleic acid content in the oil on a population, the study demonstrated that the antioxidant capacity of the oil was comparable to that of virgin olive oil, which was mainly due to the presence of tocopherols and tocotrienols. This provides an opportunity to look for alternatives for the recovery and concentration of these compounds for application as ingredients of natural origin in the development of functional foods.

To promote RPO consumption, studies that were conducted by Stuijvenberg *et al.* (2001) used RPO as an ingredient in the development of cookies for fortification programs to obtain functional cookies that were good sources of provitamin A carotenoids. All these studies have something in common: They demonstrate the nutraceutical effect of RPO on health. However, it is necessary to continue investigating the associated mechanisms for the delivery of these compounds and to protect these compounds in foods to extend their shelf lives by considering the conditions to which the foods are exposed during their manufacture. For this, microcapsules (with a range of 1 to 1000 µm) are an efficient option. They protect the compounds of interest from environmental conditions such as heat, moisture, air, and light (Comunian *et al.*, 2021) while guaranteeing regular interactions between the external and internal parts of the microcapsule and avoiding the loss of biological activity of the compounds (Thakur *et al.*, 2017). For β-carotene from various sources, microcapsules with guar gum, cassava starch, kappa carrageenan, carboxymethyl cellulose, isolated whey protein, casein alginate (Thakur *et al.*, 2017), maltodextrin, chitosan, and arabic gum (Bonilla-Ahumada *et al.*, 2018) have been explored.

CONCLUSION

Microfiltration and ultrafiltration processes have been used as low-energy alternatives within oil refinement processes (degumming, deacidification, and oil/water separation), and rarely, nanofiltrations have been explored for this purpose. This article shows the versatility of nanofiltration as an alternative for recovering phytonutrients with important biological activities, especially from CPO, which constitutes one of the essential phytonutrient vegetable sources of carotenoids, tocopherols, and tocotrienols. These compounds can be recuperated, concentrated, and used in various applications, *e.g.*, as natural ingredients for food formulations such as functional food formulations, as natural pigments in the food industry, in pharmacological, nutraceutical and cosmetics applications. Hence, this topic should be explored further to find new applications of this technology in agroindustrial chains to help generate added value.

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