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Current advances in membrane processing of wines: A comprehensive review

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ABSTRACT

Background: Membrane-based operations, especially pressure-driven membrane operations, are today well-established procedures for various applications in the wine industry thanks to their intrinsic properties and undoubted advantages over traditional methods. Emerging membrane processes, such as pervaporation, electrodialysis and osmotic distillation, forward osmosis, membrane contactors, offer new and interesting perspectives to improve quality and develop new products without compromising organoleptic properties.
Scope and approach: This review provides a comprehensive overview on the use of membrane operations in wine processing. A bibliometric and scientometric analysis has been done to provide the current advances dealing with the application of these operations in different steps of wine manufacture, including clarification, stabilization, concentration, acidification, deacidification and dealcoholization. The current challenges and perspectives are highlighted to guide further advancements of membrane technology in this field.
Key findings and conclusions: The use of conventional and emerging membrane systems offers interesting opportunities to improve and optimize current practices of the wine processing industry. Considerable progress has been done concerning the development of low-fouling materials, identification of wine molecules responsible for membrane fouling and methods to mitigate such phenomenon in the clarification of wines by microfiltration membranes. Technological progress in electrodialysis makes this process a very attractive method for tartrate stabilization, acidification and deacidification of wines. Different conventional and emerging membrane processes offer valid post-fermentation strategies to remove ethanol in wines while preserving their original characteristics. The global results provide interesting perspectives for a wider implementation of membrane processes in the winemaking industry and to redesign the traditional vinification process under the process intensification strategy.

1. Introduction

According to recent reports, the wine worldwide production is approximately estimated 244 million hectoliters (OIV report, 2023). From ancient times until now, wine is a unique and integral beverage and part of the culture of several countries. During winemaking, grape must and wine undergo several physical, chemical and biological changes resulting in a very complex composition. This latter can be divided into three main categories depending on the molecular diameter: i) solute molecules (<1 nm) such as ethanol, glycerol, sugars, organic acids, ions and monomeric phenolic compounds; ii) molecules with colloidal behaviour (between 1 nm and 1 μm) like polysaccharides, polyphenols and polysaccharides; iii) particles (>1 μm) such as microorganisms, organic precipitates and tartrate crystals. Additionally, wine contains gases (O_2 and CO_2) and a wide range of aromatic compounds.

To meet standards, distributors' requirements and consumers' expectations, winemakers used a wide array of technologies and processes to clarify, stabilize and correct the chemical composition of wines without affecting the most common organoleptic and physicochemical properties of this product (Radeka et al., 2022). Traditionally, techniques, such as addition of rectified concentrated must, addition of tartaric acid and chaptalization, are used to rectify the sugar and acid content of grape musts. For must and wine clarification and stabilization, winemakers employed traditional techniques, such as

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sedimentation, racking, conventional filtration techniques as diatomaceous-earth filtration, centrifugation, flotation, finning and chilling. However, these techniques present various disadvantages which include: i) high costs in terms of energy consumption, chemical products and labors; ii) poor propensity to comply with environmental requirements; iii) undesirable changes in the chemical composition of the product.

Despite the long historical past, the wine industry faced a high number of challenges and obstacles leading to an increasing commitment to innovation and sustainability of the entire production chain. Among these innovations, membrane technologies and processes were developed as alternative solutions to conventional techniques in order to answer several challenges concerning the stabilization and clarification of wines, the adjustment of the chemical composition of musts and wines, the control of the alcohol content and to promote the development of both sustainability and circular economy in the wine industry.

Membrane processes, based on the use of physical separation barriers (so-called membranes), are well-established technologies in wine processing at industrial scale. Nowadays, microfiltration (MF) stands out as the mandatory step for clarification and microbial stabilization in wine industry, while ultrafiltration (UF) contributes to selectively adjust the content of specific compounds that provide aroma, flavor, tannin and coloring properties while retaining chemical balance in the wine (Sui et al., 2021). Nanofiltration (NF) and reverse osmosis (RO) represent useful approach in must concentration and for controlling alcohol-content in wines (El Rayess & Mietton-Peuchot, 2016). Differently from pressure driven processes, electrodialysis (ED) acts a feasible, fast and chemical-free technology for tartrate stabilization or wine acidification and deacidification (Tsygurina et al., 2022). Osmotic distillation and pervaporation, which are driven by a vapor pressure difference, are used for the wine dealcoholization. The main characteristics of the membrane processes used in the wine industry are illustrated in Table 1.

In this comprehensive review, a deep bibliometric and scientometric analysis has been done to provide the current advances dealing with the application of membrane processes (including pressure-driven membrane processes and ED) for the treatment of wine. Apart from that, a particular emphasis has been placed on the fundamentals of membrane clarification and the main constrain (i.e., membrane fouling) occurring during the treatment via membrane technologies. Finally, important aspects and advances on controlling tartrate stabilization, acidification/deacidification of wines via ED treatment as well on the alcohol removal by membrane-based operations are also addressed.

2. Scientometric and bibliometric analysis

2.1. Methodology

This paper collects and synthetizes the available research on different membrane processes used in wine industry for different applications, including clarification, stabilization, deacidification and dealcoholization, resulting in the generation of mapping and visualization of different parts of the bibliometric records.

The Scopus database was used to retrieve the data because it is the largest tool of peer-reviewed literature with high accessibility and superior processing capabilities. The Scopus database was searched for bibliometric data in May 2023 using the following keywords: (wine) AND (membrane process) OR (crossflow microfiltration OR microfiltration OR electrodialysis OR ultrafiltration OR nanofiltration OR reverse osmosis OR membrane contactor OR membrane bioreactor). The PRISMA guidelines were used for data refinement where the total number of primary searches was 512; after filtering the documents, the final number of relevant articles was 192.

Three indicators (productivity, relevance impact and connections) established by Durieux and Gevenois (2010) were considered for this study. Variables, such as authors, institutions, countries, keywords and subject areas, were analyzed.

The resulted documents were stored in CSV format for further assessment to be analyzed. R software v. 4.2.2. (biblioshiny function) and VOSviewer.v.1.6.18 were used to perform scientific mapping by constructing bibliometric network. This analysis was complemented by the analyze search results service from Scopus.

2.2. Results

2.2.1. Evolution of publication

The trends of the number of publications per year and the cumulative number of documents are shown in Fig. 1a. The first article on membrane processes applied to wine in the Scopus database was published in 1981. During the 80's, only 2 publications were registered. It is clear that after the year 2000, the number of publications per year increased despite some fluctuations. The peak number of publications (17) occurred in 2019, indicating a growing focus on membrane processes within the wine industry in recent years.

The cumulative number of publications versus time showed a sigmoidal trend with $R^2 = 0.97$ (R^2 being the coefficient of determination which is a statistical measure representing the fitting of a regression model with the actual data). This suggests that number of publications experienced a rapid growth.

According to the outputs gained from Scopus, original articles are the most represented documents published on the use of membrane

Table 1
Characteristics of membrane processes used in wine industry (Castro-Muñoz, Ahmad, Malankowska, & Coronas, 2022; Lalia et al., 2013; Cassano et al., 2020; Castro-Muñoz, 2019, El Rayess et al., 2011).

Membrane process	Driving force	Pressure range	Pore diameter	Membrane material	Wine components retained
Microfiltration	Pressure gradient	0.5–3 bar	0.1–10 µm	Symmetrical or asymmetrical polymeric or ceramic membranes	Particles, microorganisms, colloidal aggregates
Ultrafiltration	Pressure gradient	0.5–10 bar	0.005–0.1 µm	Asymmetrical polymeric or ceramic membranes	Polysaccharides, polyphenols, proteins
Nanofiltration	Pressure gradient	2–40 bar	0.001–0.005 µm	Asymmetrical polymeric or thin-film composite membranes	sugars, some organic acids, monomeric phenolic compounds
Reverse osmosis	Pressure gradient	10–100 bar	<0.001 µm	Thin-film or dense composite or polymeric membranes	Ions
Electrodialysis	Electric field		<0.001 µm	Polymeric films with anionic or cationic exchange groups	Charged small molecules as ions and organic acids
Osmotic distillation	Vapor pressure gradient		10–40 nm	Hydrophobic polymeric hollow fibers	All compounds except ethanol and volatiles
Pervaporation	Vapor pressure gradient	Vacuum pressure between 0 and 20 mbar	30–60 nm	Dense hydrophobic polymeric membranes	All compounds except ethanol and volatiles

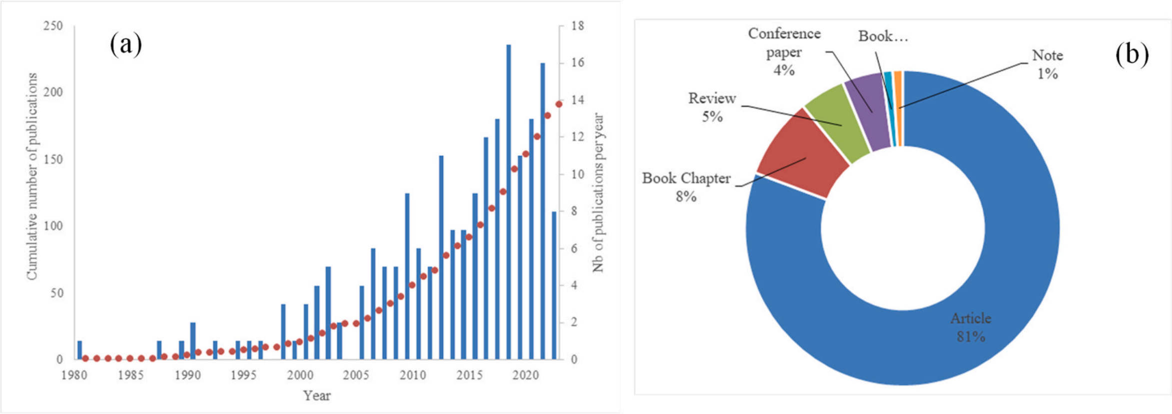

Fig. 1. (a) The annual and cumulative distribution of Scopus-indexed research articles on membrane processes in wine industry; (b) Distribution of the type of documents published on the studied topic.

processes in the wine industry and constitute 81% of the total documents, while book chapters ranked 2nd (8%). The data displayed that reviews and conference papers are 5% and 4% of the total documents (Fig. 1b).

2.2.2. Keywords analysis and clustering

The analysis of the keywords allows for determining the field of research to which the paper belongs and to determine the most studied topics. Fig. 2 illustrates the keywords, their density and their co-occurrence network. The 10 most occurred keywords are: wine, nanofiltration, microfiltration, membrane, dealcoholization, reverse osmosis, fouling, phenolic compounds, ultrafiltration and electrodialysis. As represented in Fig. 2, five keywords' clusters can be identified as follows:

- Cluster 1 (green): this cluster focuses on the MF applied for the clarification of musts and wines mainly the crossflow MF. The studies in this cluster deal with membrane fouling by wine constituents and the identification of the wine molecules responsible of fouling phenomena. The impact of the process parameters, such as transmembrane pressure (TMP), crossflow velocity, higher turbulence and membrane material, were also evoked in this cluster.
- Cluster 2 (purple): this cluster deals with ED process for potassium tartrate stabilization in wines. The main articles included in this

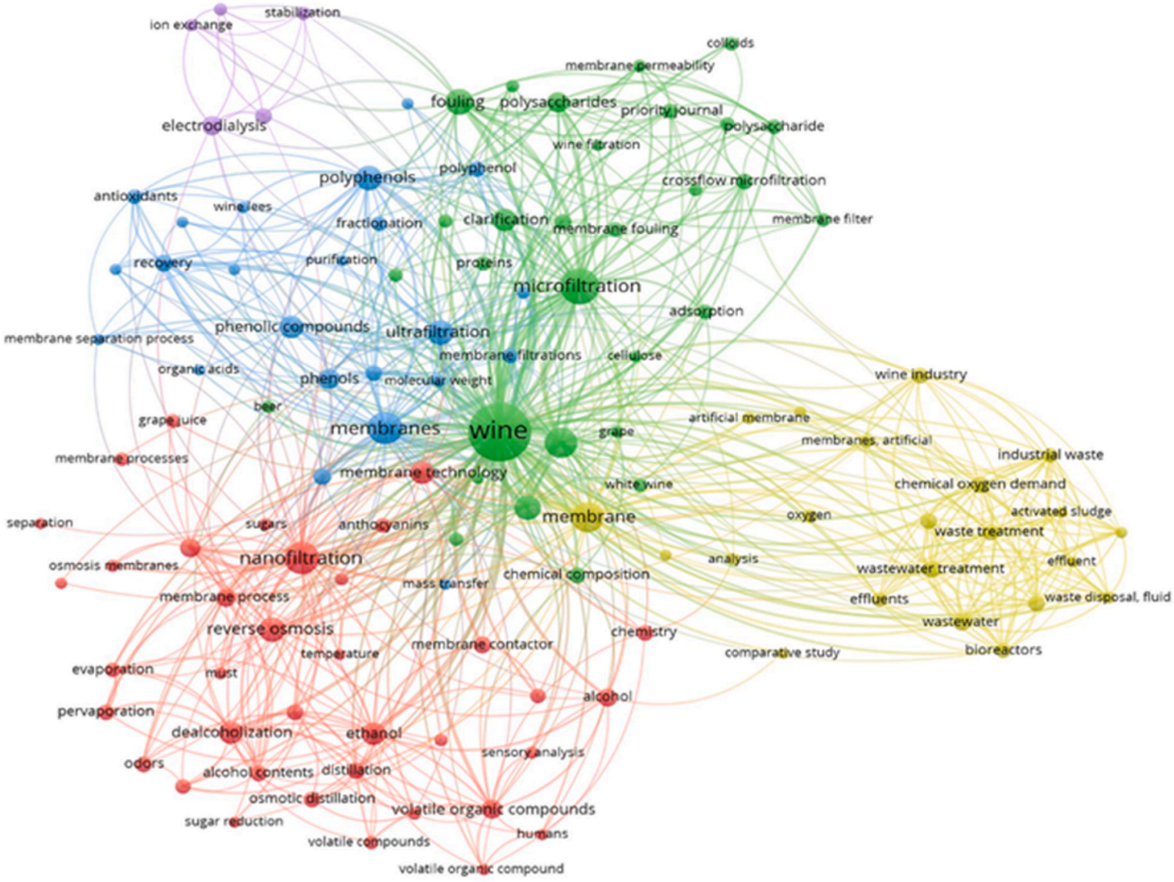

Fig. 2. Co-occurrence network of keywords.

cluster cover the impact of ED on tartaric stabilization and wine quality. They also examine the fouling problem occurring during the treatment. The wine acidification and de-acidification process by bipolar ED are also considered in this cluster.

- Cluster 3 (blue): this cluster includes keywords related to the recovery of bioactive compounds from wine wastes and by-products by membrane processes. It specifically deals with the UF process for the fractionation and purification of phenolic compounds.
- Cluster 4 (red): this cluster considers all the membrane processes used for partial or total dealcoholization of wines. RO, NF and membrane contactors are the main processes studied for dealcoholization. The studies raise the efficiency of these processes for ethanol removal and their impact on the volatile fraction (aromas) of the treated wine. They also deal with sensory studies and consumers' perception towards dealcoholized wines.
- Cluster 5 (yellow): This cluster covers the wine waste treatment by membrane bioreactors. The main focus of the studies in this cluster is the impact of the process operating parameters on the efficiency of chemical oxygen demand (COD) removal.

This statistical method taps into literature-driven insights, unveiling publication patterns and potential applications as tool to spread knowledges on the use of membrane technologies in the wine industry. This analytical approach allowed the identification of 5 clusters based on the application of membrane operations in the wine industry. Therefore, clusters 1, 2 and 4 will be developed lately in this paper.

3. Membrane processes for wine treatment

3.1. Clarification of wines and musts by cross-flow microfiltration/ultrafiltration

Cross-flow MF and UF represent well-established technologies for wine and must clarification. These processes offer the following significant advantages over conventional clarification techniques (i.e. static defecation, centrifugation, depth-filtration, diatomaceous-earth filtration):

- Combination of the clarification and microbial stabilization in a single operation instead of multiple conventional treatments leading to wine with a turbidity less than 2 NTU and less than 1 Colony Forming Unit (CFU) per bottle (Vernhet, 2019).
- Cost reduction by reducing or eliminating the use and treatment of diatomaceous earth, the multiple conventional treatments, labor costs, energy and operation time.
- Continuous and automated process with data recovery.
- Environmental considerations by reducing waste generation and saving energy (El Rayess & Mietton-Peuchot, 2016).

MF membranes, with pore size of 0.2 μm, are typically used for the clarification of red wines. These membranes can ensure microbiological limpidity and stability in a single operation producing wines with low turbidity (less than 1 NTU) without affecting their organoleptic characteristics (Daufin et al., 2001). For white wines, MF membranes with pore size, ranging from 0.1 to 0.22 μm, are typically used. These membranes produce clarified wines with turbidities lower than 0.5 NTU and more stable filtration fluxes, as the sizes of their pores are considerably smaller than the sizes of the most abundant particles in wines. Among different polymeric MF membranes (based on PES, cellulose acetate, PP, nylon) with pore size ranging from 0.2 to 5 μm, cellulose acetate membranes with pore size of 0.2 and 0.45 μm were considered the most suitable membranes in terms of physico-chemical properties of clarified red wine and permeate flux reduction (Urkiaga et al., 2002). The obtained results were attributed to the hydrophilic nature of these membranes which led to lower adsorption of polyphenols (especially anthocyanins) and polysaccharides when compared to other tested membranes. In general, most of the membranes exhibited similar turbidity reduction; however, PES membranes (0.45 μm) needed the longest filtration time, although their initial permeate fluxes were found as the highest ones. The authors suggested that back-flushing, back shocking, or infrasonic pulsing could be implemented to reduce the fouling, which is commonly provoked by polysaccharides (Urkiaga et al., 2002).

The limpidity of red monovarietal wines clarified with ceramic membranes of 0.2 μm resulted higher than that of the same wines clarified with egg albumin, or by progressive clarification with egg albumin, followed by filtration plates on cellulose (Martínez-Lapuente et al., 2017). In particular, for all wines investigated the total polyphenol index was reduced from 13.4 up to 20.5%. The MF process also produced the highest retention of polysaccharides and proanthocyanidins, especially polysaccharides rich in arabinose and galactose, yeast mannoproteins and highly polymerized phenols.

Discriminant analyses revealed that the content of total monosaccharides forming polysaccharides is the main differential factor between cross-flow MF and other clarification processes.

According to McRae et al. (2017), the average particle size of red wines decreased significantly with cross-flow MF. In particular, the concentration of polysaccharides in Cabernet Sauvignon wines decreased after filtration with 0.45 μm PES or nylon membranes while maintaining tannin and color. This decrease in the average particle size is crucial for wine limpidity and clarity since large molecules, such as polysaccharides and microorganisms, can contribute to the haziness state of the wine, which alters the presentation and degustation of the wine. The global results indicated that commonly applied MF practices do not affect wine color and have a minimal effect on the sensory profiles of red wines.

UF membranes are also available for wine clarification (Goncalves et al., 2001). However, such fine clarification leads to the removal of an important part of the colloidal matter (Oberholster et al., 2013), leading to the decrease of wine flavour intensity and, accordingly, to a deterioration in quality.

Recently, Sui et al. (2022) investigated the use of UF in the treatment of white and rosé wines on an industrial scale to remove phenolic compounds associated with astringency. UF was performed by using cross-flow filtration systems fitted with spiral-wound PES membranes having a MWCO of 5 and 10 kDa. For all treated wines, the fractionation was higher than 90%. Wine macromolecules, such as phenolics and proteins, were substantially retained by the UF membranes while other compositional parameters such as pH, free and total SO_2, volatile acidity and viscosity were not significantly affected. Chemical analysis also revealed the rejection of low MW molecules by fouled membranes, confirming that membrane selectivity is not only associated with size exclusion phenomena (Galanakis et al., 2013).

3.1.1. The principle of cross-flow microfiltration

In cross-flow MF the fluid to be filtered flows tangentially to the membrane surface and permeates through the membrane due to a pressure difference. When the feed flows over the membrane surface, the filtrate (known as permeate) goes through the membrane while the concentrate (known as retentate) is accumulated. This particular tangential flow generates shear forces at the membrane surfaces, resulting in less membrane fouling when compared with dead-end filtration. The pressure gradient across the membrane would force solvent and smaller species through the membrane pores while the larger molecules are retained. Specific membrane modules, such as flat, spiral-wound, and hollow fiber, can be installed and operated easily.

To some extent, the produced shear forces are not high enough, and consequently, some low molecular weight solutes tend to deposit on the membrane surface, together with a layer of concentrated solution. Thanks to the convective flow pattern, the concentration of the feed bulk solution at the surface interface (C_w) increases as a function of operating time (see Fig. 3a), resulting in a gel formation with a concentration (C_g).

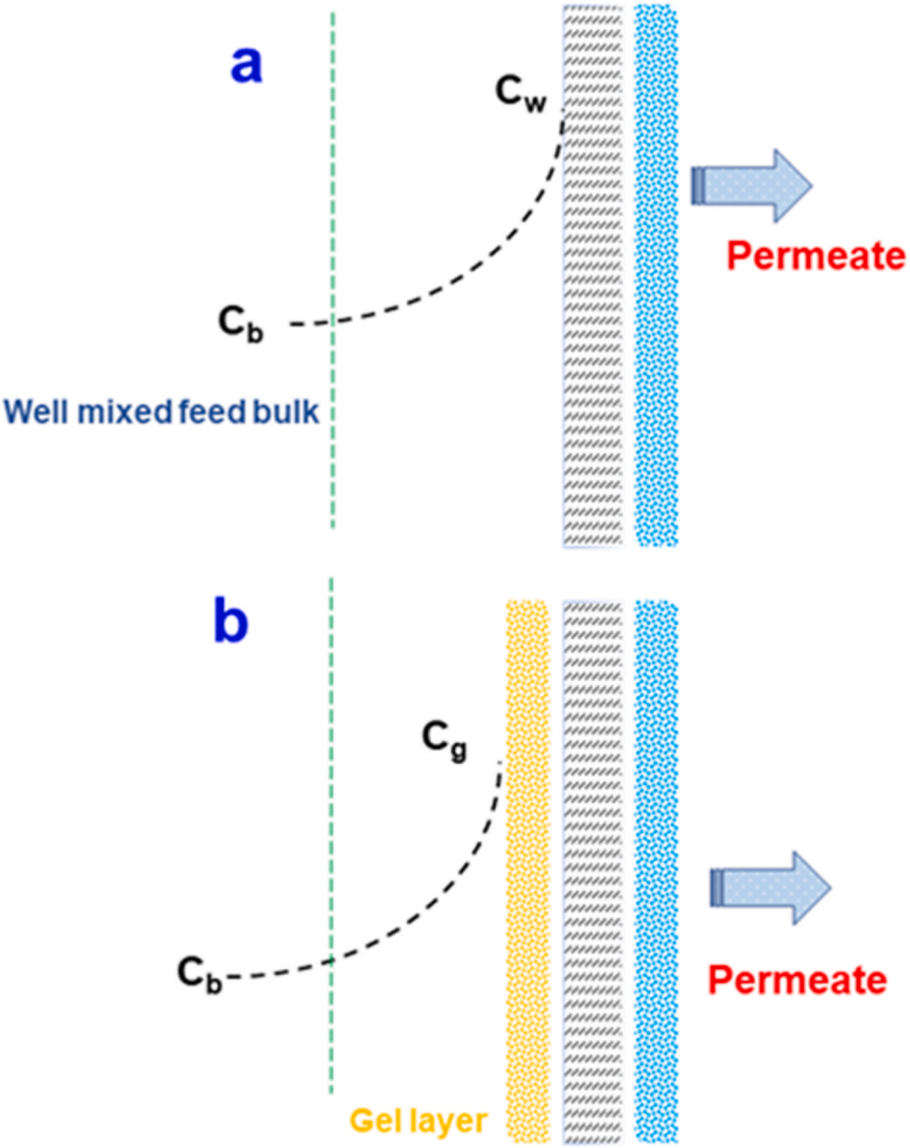

Fig. 3. General scheme illustrating concentration polarization in membrane filtration. (a) Concentration profile before gel formation; (b) concentration profile with a gel layer formation at the membrane surface.

This latter concentration is higher than the feed bulk concentration (C_b), as illustrated in Fig. 3b. Such a phenomenon is well-known as concentration polarization. When C_g is higher than C_w, the total flux increases when there is pressure drop increase, while when C_w is equal to C_g, any increase of TMP results in a compaction of the polarization layer and consequently increases its thickness (Murkes, 1990). Therefore, the resistance indeed increases simultaneously with pressure but no flux increment is observed. Typically, the concentration polarization phenomenon also results in a flux decay as a function of operating time. Generally, the flux declines fast at the beginning of the operation, and subsequently, when equilibrium conditions are achieved, it is offset by only a slow decline. This slow flux decay is credited to the granulometry of the deposited layer, as it is continuously enriched with finer and colloidal fractions (Ilias & Govind, 1993).

3.1.2. *Fouling problems in wine cross-flow microfiltration*

The use of MF and UF in wine processing is greatly affected by membrane fouling due to the deposition and accumulation of colloidal particles and macromolecules on the membrane surface or within membrane pores. This phenomenon can cause severe flux drops affecting the economic viability of the process, as well as an excessive retention of some components, which may affect the product quality.

Yeast, bacteria, cell debris and typical wine macromolecules such as polyphenols, polysaccharides and proteins are typical foulants of MF membranes (Boissier et al., 2008). Polysaccharides and polyphenols were identified as the main foulants of inorganic tubular alumina MF membranes used for the clarification of red wine (Belleville et al., 1992).

3.1.2.1. Fouling mechanisms in wine cross-flow microfiltration. During wine filtration, permeate flux declines over time. It is characterized by a rapid initial drop, followed by a long-term gradual decrease, and ending with a steady-state flux. As for other fermented food products, membrane fouling in wine filtration can be divided into: a) *internal fouling* due to adsorption and pore plugging of small particles and colloids within the internal structure of pores; b) *external fouling* by particles, macromolecules and macromolecules aggregates causing pore blocking and cake formation (Belfort et al., 1994). More specifically, three different mechanisms can contribute to this phenomenon: concentration polarization quickly followed by cake layer formation, (ii) adsorption of solutes onto the membrane surface and pore walls, and (iii) blockage of pores (El Rayess et al., 2011a).

Fouling mechanisms can be better identified and described using four empirical models or blocking laws. The four models are based on the laws only valid for constant pressure filtration and can be described by a common mathematical equation:

$$\frac{d^2t}{dV^2}=k.\left(\frac{dt}{dV}\right)^n \tag{1}$$

The representation of this equation in logarithmic scale directly gives the value of the blocking index (n) and the resistance coefficient (k). Depending on n value, the model can be identified as follows: cake filtration ($n = 0$), intermediate blocking ($n = 1$), standard blocking ($n = 3/2$) and complete blocking ($n = 2$).

The type of fouling can be also identified by the resistance-in-series model, determined by the Darcy law. The measurement of the total resistance (R_t) can lead to the identification of the reversible resistance (R_{rev}) where fouling can be removed without chemical cleaning and irreversible resistance (R_{irrev}) due to the adsorption of wine molecules and internal blocking of pores.

3.1.2.2. Findings on fouling during wine cross-flow microfiltration. Factors contributing to membrane fouling during cross-flow MF are summarized in Fig. 4. The complex composition of wines, including suspended solids and colloids, poses a significant challenge to membrane filtration. The presence of these components and their interactions can lead to the formation of fouling layers on the membrane surface. The main wine components responsible for membrane fouling were identified. They consist of polysaccharides, polyphenols, proteins, yeasts and bacteria (Belleville et al., 1992; Boissier et al., 2008; El Rayess et al., 2011b; El Rayess et al., 2012; Salazar et al., 2007; Vernhet & Moutounet, 2002; Vernhet et al., 1999).

Several studies have been carried out on the impact of wine constituents on the fouling behaviour of membranes during membrane filtration. The initial rate of flux decline in the MF of crude wine with capillary PES membranes with an average pore size of 0.1 μm, as well as the flux evolution, is governed by fine particles such as colloidal size-range aggregates and dissolved constituents with hydrodynamic diameter lower than 2 μm (Vernhet et al., 2003). These compounds can penetrate into the membrane pores leading to internal fouling phenomena. On the other hand, the overall membrane resistance during wine filtration is governed by external fouling due to the polarization of retained species on the membrane surface. This type of fouling is considered the dominant fouling mechanism in the MF of crude wine: it occurs within the first steps of filtration (minutes or even seconds) and it is greatly enhanced by the TMP. The external fouling was shown to be mainly reversible since the polarization of the retained species did not lead to the formation of a structured cake, even for high operating pressures. Boissier et al. (2008) analyzed the particle deposition (i.e. *Saccharomyces cerevisiae* yeast and fines such as lactic bacteria and colloidal aggregates) in the cross-flow MF of red wines with capillary polypropylene (PP) membranes having a pore size of 0.2 μm. Yeasts formed reversible deposits in relation to their surface properties. The flux evolution at different TMP values indicated that a limiting permeation flux was reached, and that significant membrane fouling can be avoided by maintaining the flux under a TMP value of 0.4 bar. Importantly, fines formed an adherent and irreversible cake above a given TMP. When yeast and fines were processed together, the deposit formation and the structure resulted strongly dependent on the TMP stepping mode.

El Rayess et al. (2012) investigated the impact of wine colloids such as tannins, pectins and mannoproteins on fouling phenomena of multichannel ceramic membranes with an average pore diameter of 0.2 μm in the cross-flow MF of red wine. A quick adsorption, followed by the formation of aggregates causing pore blocking and cake formation, was considered the main mechanism of fouling due to tannins. In particular, pectins caused the formation of a gel-type layer which was found to be compressible under high pressures. Different fouling mechanisms were identified in the presence of mannoproteins; however, the end of filtration was governed by a cake layer filtration mechanism. In fact, colloids are unstable and have changeable properties depending on their volume fraction and concentration. During filtration, transitions between the different phases of colloids (gas, liquid aggregate, gel and solid) can occur at the surface of the membrane due to the volume fraction of the retained compounds increases leading to intermolecular interactions through van der Waals interactions and subsequently aggregation.

Recently, Rosária et al. (2022) evaluated the impact of cross-flow MF

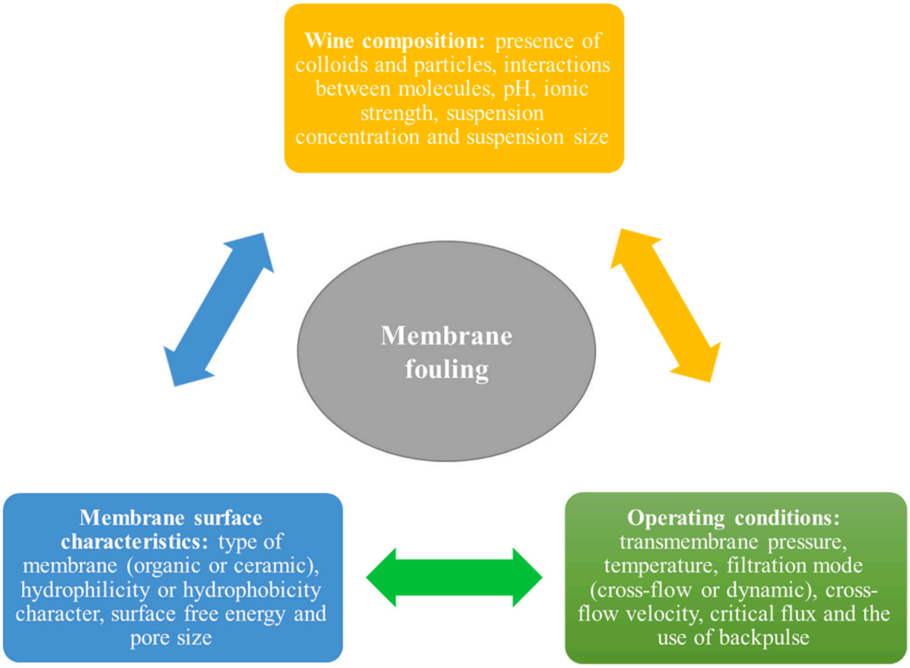

Fig. 4. Factors influencing the membrane fouling during cross-flow microfiltration of wines.

and membrane plate filtration carried out under winery-scale conditions on the turbidity level, phenolic composition, chromatic characteristics and sensory profile of red wine. They pointed out that the two types of filtrations did not have a clear impact on the sensory characteristics of the treated wines. The global results indicated that the initial wine composition had a greater impact on the wine characteristics and process performance if compared with the filtration process itself.

The membrane material plays a crucial role in controlling membrane fouling during wine cross-flow microfiltration. According to Ulbricht et al. (2009), the adsorption of polyphenols and polysaccharides on polymeric membranes is strongly influenced by the membrane material. In particular, individual polyphenols and polysaccharides were only marginally adsorbed by a capillary PP membrane of 0.2 μm but strongly adsorbed by PES membranes with the same porosity. The adsorption of polyphenols on PES membranes was attributed to polar interactions such as van der Waals interactions and electron donor-acceptor interactions and multiple hydrogen bonds towards the additive polyvinylpyrrolidone (PVP) used in membrane manufacture. The presence of aggregates between polyphenols and polysaccharides in red wine had a major contribution to adsorptive fouling which resulted strong for PES membranes but very weak for PP membranes. Schroën et al. (2010) confirmed that PES membranes bind significantly fewer tannins than membranes made from PVP. The different tannin-binding capacity of filtration membranes was attributed to differences in the surface properties. Intermolecular adhesions between proteins and membranes appear to be an important component of flux decline. Koehler et al. (2000) reported that the use of hydrophilic surfaces minimizes the adhesion of proteins and hence membrane fouling. Polysulfone (PS) membranes were modified by grafting with 2-hydroxyethyl methacrylate (HEMA) after low-temperature plasma treatment to increase their hydrophilicity. These membranes exhibited lower contact angle and reduced both adhesion forces and adsorbed amount of proteins (lysozyme) when compared to unmodified PS membranes. Above the isoelectric point of the protein small adhesion forces between the protein and the hydrophilic polymer were detected. Adversely, at the pI of the protein lysozyme−lysozyme aggregates are more likely to form causing plugging of membrane pores due to convective drag. The impact of wine polysaccharides and polyphenols on membrane fouling of hollow-fiber membranes with different polarity was also investigated by Vernhet and Moutounet (2002). Sharp flux declines identified within the first minutes of the process were attributed mainly to physico-chemical interactions of membrane/wine constituents and wine constituents/wine constituents in the membrane's outermost pores and on its surface, promoted by the local hydrodynamic conditions. Polysaccharides played a prominent role in flux decline independently by the pore size distribution and polarity of the used membrane. On the other hand, membrane polarity was found to strongly affect fouling phenomena due to polyphenols. Differences in terms of permeate flux between selected membranes were mainly attributed to their different pore size distribution.

As shown in Fig. 4, operating conditions also play an important role in membrane fouling. Cassano et al. (2008) analyzed the effect of operating conditions (temperature, feed flow rate and TMP) on membrane fouling in the clarification of grape must with PS membranes of 100 kDa in hollow fiber configuration. Fluxes decays were analyzed through the resistance-in-series model. Cake layer resistance (R_c) and fouling resistance (R_f) increased with TMP due to the enhanced flux and convective flow of the solute towards the membrane. For TMP values higher than 0.4 bar the contribution of R_c to the total resistance (R_t) was higher than R_f. To some extent, R_c decreased by increasing the feed flow rate due to the enhancement of the mass transfer coefficient and the reduction of the concentration polarization layer. An increase in temperature in the range of 15–23 °C produced a significant decrease of the cake layer resistance due to back diffusion of solutes into the bulk solution, while a slight increase of both cake layer and fouling resistances was observed in the range of 23–39 °C.

Palacios et al. (2002) investigated the effect of TMP on the permeate flux at several feed flow rates in the clarification of sherry wines and brandies with a filtration cell equipped with polyvinylidene fluoride (PVDF) membranes having a pore size of 0.45 μm. A constant ratio between the optimum TMP and the feed flow rate of about 32×10^{-4} Pa L^{-1} h^{-1} was identified for all the treated wines. For a feed flow rate of 360 L h^{-1} the estimated optimum pressure resulted in 1.1 bar, for all products. The average size of the particles in the gel layer was calculated to be in the range 10–50 nm suggesting that molecules from small proteins to nucleic acids, large globules of polymers, or cell components can contribute to its formation according to their molecular weight. The physico-chemical stability of sherry wines clarified by cross-flow MF resulted higher than that obtained by conventional filtration.

The concept of critical flux in MF of colloidal suspensions was, for the first time, introduced in 1995 (Zhang et al., 2015). It was considered as the flux below which a decline of flux with time does not occur, while fouling is observed above it. According to El Rayess et al. (2011b), this definition is not appropriate for wine filtration since the adsorption of wine colloids on membrane materials occurs frequently even in static conditions. Indeed, the critical flux was considered as the flux above which an irreversible deposit appears at the membrane surface. Authors studied the critical fouling conditions during wine cross-flow MF with a multichannel ceramic membrane of 0.2 μm to limit fouling caused by wine colloids and improve process performances. Experiments were performed with filtered red wine added with different concentrations of colloids. It was found that irreversible fouling occurred from the beginning of the filtration and even at low pressures. Adsorption on membrane material and formation of deposit layer were found to be the main mechanisms of fouling with filtered wine and wine added with mannoproteins, respectively. A loss of average flux beyond a given limit of TMP was observed for filtered wine containing mannoprotein and pectin. It was attributed to the compaction of a gel layer. El Rayess et al. (2011b) also introduced the concept of 'threshold flux' in wine MF defining a new fouling criterion based on the ratio between irreversible resistance and hydraulic membrane resistance. When this ratio is less or equal to one, a certain degree of fouling is acceptable.

Membrane adsorptive phenomena can influence the aromatic profile and the phenolic quality of treated wines. Arriagada-Carrazana et al. (2005) found that the filtration of Cabernet Sauvignon through a two-step MF process produced a decrease of the total polyphenolic index of 10% (with tannins and anthocyanins reduced of 4.8% and 2.4%, respectively), as well a significant variation of some aromatic compounds. Similarly, Buffon et al. (2014) found that one sensory attribute and six sensory attributes were significantly modified in the MF of white and red wines, respectively, with a PES membrane having a pore size of 0.22 μm.

3.1.3. *Latest advances in wine cross-flow microfiltration*

It is well known that high fluid velocities in cross-flow MF allow to induce high shear rates at the membrane surface so limiting the cake formation on the membrane surface. These high velocities generate large axial pressure gradients that in turns cause large pressure drops along the membrane which reduces the mean TMP and therefore the mean flux. Physical cleaning methods, such as back-flushing, back-washing and back-pulsing, are typical methods of operation which allow to remove reversible foulants from the membrane surface by inverting periodically the permeate flux through the membrane through the use of a secondary pump, thereby lifting foulants from the membrane surface and reducing concentration polarization near the membrane surface (Gao et al., 2019). These steps are time consuming and add mechanical stress to the filtration devices which in turns result in a loss of both capacity and efficiency of the equipment.

Dynamic filtrations represent very useful approaches to improve the membrane performance in wine applications. These systems are based on the use of mechanical devices devoted to promote turbulence at the membrane surface so to produce high shear rates independent on the feed flow which can be kept at low values. As a consequence, higher permeate fluxes can be obtained in comparison to cross-flow filtration systems, together with excellent transmission of microsolutes due to the absence of a particle layer on the membrane (Fillaudeau et al., 2007). A comparison between dead-end, cross-flow and dynamic filtration is illustrated in Fig. 5. Dynamic filtration modules use either vibrating or rotating membranes or a mechanical device with a rotating or vibrating disc close to the membrane surface (Jaffrin, 2012).

The first rotating dynamic cross-flow filter introduced into the wine industry was patented by TMCI Padovan in 2011 (TMCI, 2011). It is known as multi-shafts system with rotating ceramic membranes and consists of two stacks of rotating membranes that rotate counter-current to create the maximum shear forces at the membrane surface. This configuration can increase the permeate flux significantly.

El Rayess et al. (2016) studied the impact of membrane material, wine composition and operating conditions on the performance of a shear-enhanced process used in the clarification of both filtered and crude simulated wine. It was based on the use of a Rotating and Vibrating Filtration (RVF) module constituted by 3 flat blade impellers in a confined cell. For filtered wine, the mechanical impact of RVF resulted quite limited and irreversible fouling was dominant. High fluxes (>1000 L h^{-1} m^{-2}) were obtained in standard operating conditions for both hydrophilic and hydrophobic membranes (PES and PTFE membranes of 0.2 μm, respectively). On the other hand, the mechanical effect of the RVF membrane was relevant with crude simulated wine and the increase in permeability for PTFE and PES membranes was of 34% and 300%, respectively. In this case, the establishment of irreversible and reversible fouling resulted strongly dependent by interactions between membrane material and feed solution. In particular, for PTFE membranes fouling was greatly affected by molecules/membrane interactions; for PES membranes fouling was mainly affected by the hydrodynamics of the system.

Silicon carbide (SiC) membranes are increasingly used for membrane manufacturing thanks to their higher stability, longer lifetime and better propensity to cleaning/sterilizing processes in comparison to synthetic membranes. These membranes provide improved permeate fluxes at low pressure and minimal fouling when used for filtration of cloudy liquids such as wine (Hofs et al., 2011). Trevisan et al. (2020) found that SiC membranes with pore sizes of 0.25 μm and 0.60 μm (Crystar® FT250 and FT600, respectively) exhibited higher fluxes (up to 2.5 times) and greater fouling resistance in the treatment of both red and white wines when compared with conventional 0.10 μm oxide ceramic membranes. These membranes provided an efficient retention of microorganisms without affecting color and organoleptic characteristics of the treated wines. These results have also been recently confirmed on an industrial scale on viscous high-sugar matrices such as unfermented grape juice and lees (Trevisan et al., 2022).

3.2. Grape must concentration by nanofiltration and reverse osmosis

When the climate conditions do not allow the optimal ripening of grapes, the consequences will be severe on the quality of must and consequently on wine. The resulting musts will show insufficient sugar content and the wines will be low in alcohol content impacting the organoleptic quality. In the wine industry, different additive techniques are employed to adjust sugar levels in musts as chaptalization (adding cane sugar, beet sugar or corn syrup), concentrated musts, or rectified concentrated musts. These techniques lead to an increase in wine volumes and changes in the organoleptic quality of wines. Alternative techniques have been accepted by the official authorities, including NF and RO. The former one (NF) is a pressure-driven membrane process that lies between the separation characteristics of RO and UF processes (Salehi, 2014). The pore size of NF membranes is in the range of 0.5 and 1 nm. Three separations models can be identified in NF: the electrostatic interaction, the steric hindrance model and the solute-diffusion model (Yadav et al., 2022). RO is considered as diffusion-controlled process that counteracts the natural osmotic pressure generated by the difference of concentrations of two solutions, with a semipermeable dense membrane acting as a filter, allowing only specific solvent molecules (e.g., water) to pass through. By applying a pressure higher than the osmotic pressure, the solvent flow is reversed, moving from a less concentrated solution to a more concentrated one. This phenomenon leads to an increase in the solute concentration in the remaining solution. Both techniques were studied for grape must concentration by partly extracting water. Mietton-Peuchot et al. (2002) found that RO could serve as a viable alternative to chaptalization and vacuum evaporation for grape must concentration. They found that maintaining low temperatures (around 10 °C) and applying high pressure (75 bar) effectively prevented essential components of the grape must from passing through the membrane. In a study conducted by Versari et al. (2003), the focus was on evaluating the specificity of two NF membranes to concentrate grape must. Their findings revealed that these NF membranes exhibited rejection coefficients ranging from 77% to 97% for sugars. Kiss et al. (2004) proposed a combination of RO (60 bar and 20 °C) and NF (70 bar and 40 °C) to produce concentrated grape must with a high sugar concentration around 45°Brix. Gurak et al. (2010) investigated the concentration of grape juice using RO and showed that the physical and chemical properties of the concentrated grape juice increased in proportion to the volumetric concentration factor. They identified that the optimal process conditions for achieving high permeate flux values were an applied pressure of 60 bar and a temperature of 40 °C. Santos et al. (2008) conducted a study on the impact of six distinct NF membranes on the process performance for grape must concentration. Results indicated that NF200, NFT50 and NF270 membranes exhibited superior rejection coefficients for sugars (88%) compared to organic acids (37%).

Unfortunately, due to the chemical complexity and high viscosity of musts, membrane fouling is hindering the lifetime and accuracy of both processes. Pati et al. (2014) investigated the influence of operating conditions of NF and RO on the main characteristics of concentrated grape musts. They found that the applied pressure was the main parameter to obtain high-quality wine. However, increasing pressure led to an increase in membrane fouling due to liquid compressibility and an increase in membrane hydraulic resistance. Bianchi et al. (2023) demonstrated that membrane fouling occurring during grape must concentration by NF and RO is due to a complete blockage fouling

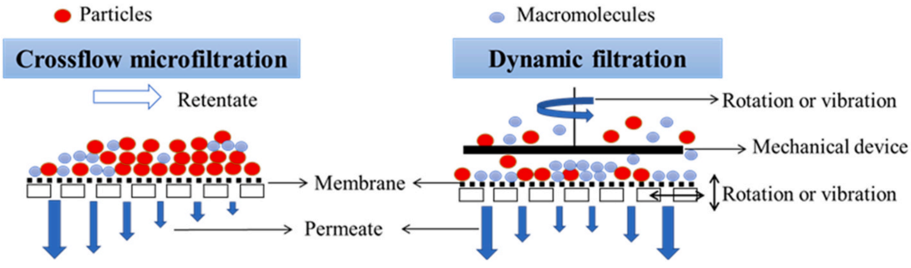

Fig. 5. Comparison between cross-flow and dynamic filtration.

mechanism. However, they showed that NF membranes exhibited higher flow rates with lower retention coefficients of sugars compared to RO. Salgado et al. (2016) studied the fouling mechanism of NF membranes used for the sugar control in grape must. They found that fouling kinetics consists of three consecutive steps: an initial pore-blocking step followed by a cake deposition phase and an increase in compression until arriving at compaction giving a slower kinetics.

3.3. Electrodialysis and bipolar electrodialysis for tartrate stabilization, acidification and deacidification of wines

3.3.1. Tartrate stabilization by electrodialysis

Among temperature instabilities, the tartrate precipitation is the most studied case in winemaking. If a wine is untreated and chilled to a low temperature in the consumer's refrigerator, the unstable tartrate crystals may precipitate out of the wine and settle at the bottom of the bottle. These crystals are unattractive to consumers who can confuse them with microbial spoilage or chemical deterioration of wine. Tartrate salts (potassium hydrogen tartrate, KHT and calcium tartrate CaT) are naturally present in grape must usually at saturated levels. In wine, they become insoluble due to the presence of ethanol and can precipitate during the storage at low temperature.

In winemaking, various techniques and methods of KHT stabilization were used. The common stabilizing methods include chilling or cold stabilization, contact processes, ion exchange and the use of chemical compounds to prevent KHT precipitation (Dabare et al., 2023). Cold stabilization is the most widely used technique for wine stabilization. It is conducted after fermentation and before bottling to prevent KHT precipitation during storage or chilling post-bottling. The wine is chilled to a temperature just above its freezing point and is held at that temperature for two or three weeks. Although cold stabilization has proven effective, it is perceived in the wine industry to suffer from some significant disadvantages including long processing time, high energy cost, and large capital investment to provide tanks and extra refrigeration capacity. Also, in red wines, polyphenol precipitation occurs during tartrate precipitation leading to a loss in wine quality. In addition, the wastes generated by cleaning tartrate-encrusted tanks may result in additional environmental costs.

Electrodialysis (ED) was developed in the wine industry as an alternative for cold stabilization due to its limitations cited before. This process is a separation or concentration process of ions in solutions. It utilizes an electric field to migrate ions from one solution to another through a semipermeable membrane. Specially, cation- and anion-exchange membranes are arranged alternately to form thin compartments called cells (El Rayess & Mietton-Peuchot, 2016). The principle of ED for potassium tartrate stabilization in wine is shown in Fig. 6. After the application of the electric field, the tartaric acid anions (TH$^-$ and T^{--}) will be attracted by the positive electrode; they will be able to pass through the anion exchange membrane. Once in the water stream, they will be blocked by the cation exchange membrane and remain in the water stream. In the opposite direction, the cations (K$^+$ and Ca^{++}) will migrate to the cathode through the cation exchange membrane. Once in the water stream, they will be blocked by the anion exchange membrane and remain trapped in the water stream. At the end of the treatment, the stabilized wine will constitute the dilute compartment while the water charged with anions and cations will constitute the concentrate or brine compartment.

To achieve the desired reduction level of the ions or the degree of deionization (DD), the wine is recirculated into the electrodialyzer. The DD is the reduction level of electrical conductivity of the wine during the treatment (El Rayess & Mietton-Peuchot, 2016). This parameter is determined before the wine treatment by ED. Several tests were developed to evaluate the tartaric instability degree of wine. The cold test reproduces the same conditions (−4 °C for 6 days) of a cold stabilization but at a lab scale. This test is time-consuming and sometimes unreliable. The saturation temperature (T_s) was also proposed as a stability indicator of the wine. The wine is considered stable when low values of T_s are obtained. Gonçalves et al. (2003) reported that the variation of the saturation temperature with the DD of white wine is linearly correlated by the equation: $T_{sat} = 20.3–0.44$ x degree of deionization. However, it has been verified that T_s test is inaccurate and non-reproducible (Lasanta & Gómez, 2012).

The mini-contact test measures the degree of tartaric instability (DTI) which is positively correlated with the reduction of the wine conductivity. In this test, the drop of conductivity is monitored in a wine enriched with 4 g/L of KHT crystals (secondary nucleation) at −4 °C for 4 h under stirring. Although these tests are simple, they result in inaccurate results (under-prediction of the wine DD) and wines need to be checked again using a more robust mini-contact test called ISTC50. Bosso et al. (2016) proposed a rapid conductimetric test for the measurement of wine tartaric stability. This test is a modified version of the mini-contact test with the following operating parameters: T: 0 °C;

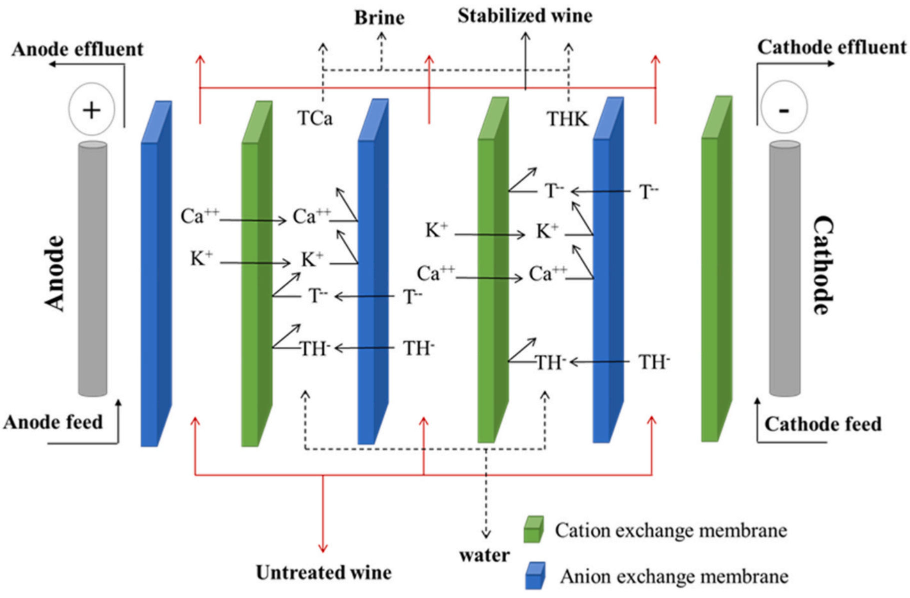

Fig. 6. Electrodialysis principle for wine tartrate stabilization (Adapted from El Rayess & Mietton-Peuchot, 2016).

duration: 4 min and dose of added KHT: 10 g/L. Henriques et al. (2019) developed a controlled freeze-thawing test for the determination of DD required for wine tartaric stabilization by ED. In this test, wines are frozen at −20 °C with controlled nucleation for 24 h and then they are thawed at 0 °C. This test allows the direct generation of the DD without using fitting curves as for the mini-test contact. Also, the authors reported that DD obtained by the freeze-thawing test is at least 5.7% higher than the one predicted by the mini-contact test.

According to Romanov and Zelentsov (2007), the ED treatment influenced tartaric acid and ions concentrations; however, no incidence was found on the organoleptic and phenolic characteristics of treated wines. When comparing the ED treatment to cold stabilization, Corti and Paladino (2016) demonstrated that both treatments did not affect the organoleptic characteristics of both white and red wines. Recently, many studies have been interested in the fouling phenomena occurring during wine ED (Bdiri et al., 2020; Pasechnaya et al., 2023; Tsygurina et al., 2022). It was shown that polyphenols, as well colloidal particles formed by polyphenols and other wine components, actively interact with ion-exchange membrane. These interactions are mainly electrostatic interactions and hydrogen bonds. Anthocyanins (with their flavylium cation form) can interact with cation-exchange membranes leading to membrane fouling. Bdiri et al. (2020) used a mixture of four solvents to extract phenolic foulants from ion-exchange membranes employed for wine tartrate stabilization by ED at industrial level; results of ultra- and high-performance liquid chromatography (HPLC) and attenuated total reflectance Fourier-transform infrared (ATR-FTIR) clearly indicated that phenolic acids, quercetin, kaempferol and anthocyanins were the main components responsible for membrane fouling through electrostatic interactions with the membranes. The negatively charged molecules or colloidal particles (proanthocyanidins, proteins and organic acids) can interact with the positive groups of the anion-exchange membrane leading to fouling. Membrane fouling during wine ED leads to a decrease in ion-exchange capacity, conductivity selectivity and mechanical stability of the membranes (Pasechnaya et al., 2023). At the end, ED offers several advantages over these traditional methods. It is a continuous process that can be operated at ambient temperature, resulting in lower energy consumption (Bories et al., 2011). It can also be used to selectively remove tartaric acid ions, which minimizes the risk of flavor alterations compared to other stabilization methods. Furthermore, the process does not require the use of additives or chemicals, which is beneficial for organic winemakers and consumers who prefer chemical-free wines.

3.3.2. *Must and wines acidification and deacidification by bipolar electrodialysis*

pH and acidity are very important parameters in winemaking. Their mismanagement may compromise the quality of wine and may have repercussions on the technology scheme used in winemaking. In fact, the physico-chemical and microbiological stabilities of wine are strictly related to pH. pH can influence the behaviour of fermentations (alcoholic and malolactic) and is directly impacting the free and macromolecular form of sulfur dioxide. It also influences the solubility of tartaric salts and the equilibrium form of anthocyanins, impacting directly the color of red wines.

Over the last two decades, the effect of global warming has been very perceptible in the wine industry. Wines have higher ethanol levels and higher pH values. The high values of pH are not only due to a lack of organic acids content but rather an excess of cations (potassium and calcium) leading to the salification form of the acids (Berbegal et al., 2019). Traditionally, several strategies have been adopted for the acidification of musts of wines leading to an increase in titratable acidity and a decrease in pH. The most used technique is the chemical acidification by tartaric acid. The European community allows a maximum acidity increase of 1.5 g/L for musts and 2.5 g/L for wines (Regulation EU 1308, 2013). In the United States, the regulation allows an increase of 9 g/L of tartaric acid in the finished wines (Electronic Code of Federal Regulations, 2017). The mastering of tartaric acid addition is not a simple matter because it depends on potassium level, ethanol strength, initial pH and the presence of protective colloids. The supersaturated potassium bitartrate wines may lead to the precipitation of potassium tartrate salts (Devatine et al., 2002). As a result, the prediction of the pH increase is quite difficult.

The bipolar ED is a physical treatment developed as an alternative technique for chemical acidification that allows the continuous extraction of potassium from the wine and the enrichment in H_3O^+ ions leading to the decrease of pH, as illustrated in Fig. 7. It is an electrochemical process that utilizes ion-exchange membranes, bipolar membranes and an electric field to selectively remove or transfer ions from a solution. Bipolar membranes and cation-exchange membranes are arranged alternately to form thin compartments called cells. The bipolar membrane is composed of three parts: an anion-exchange membrane, a cation-exchange membrane and a hydrophilic interface at their junction (Bazinet et al., 1998). Bipolar membranes carry out the dissociation of water (H^+ and OH^-) in the presence of an electric field. This leads to maintaining the acid/base ionic balance of the process. When applying the electric field, the potassium cations present in wine are attracted by the negative electrode. They pass through the cation exchange membrane and they will be found in the water compartment where they will be stopped by the bipolar membrane. The ions H^+ generated by the bipolar membrane will be found in the wine compartment to conserve the ion equilibrium while the OH^- ions will be installed in the water compartment. The extraction of K^+ ions from the wine and the injection of H^+ ions in the wine will lead to the acidification of the wine. This will result in a decrease in pH and an increase in titratable acidity (El Rayess & Mietton-Peuchot, 2021). The benefits of wine acidification by bipolar ED are:

- Precise pH adjustment: bipolar ED allows to regulate the acidity of wines with high precision (0.05 units of pH). By selectively removing the potassium ions, it enables the attainment of desired pH targets, thereby enhancing wine quality and technological properties. According to the International Organization of Vine and Wine (OIV), the maximum treatment value is 0.3 units of pH and the total increase in total acidity should not exceed 54 meq/L.
- Minimization of undesirable side effects: Traditional acidification methods including the addition of tartaric acid may introduce “harsh” or “sour” undesired flavors affecting the sensory profile of wines. In contrast, bipolar ED offers a more controlled approach minimizing the risk of flavor alteration. The process allows a better conservation and an improved aging of wines (Granes et al., 2009).

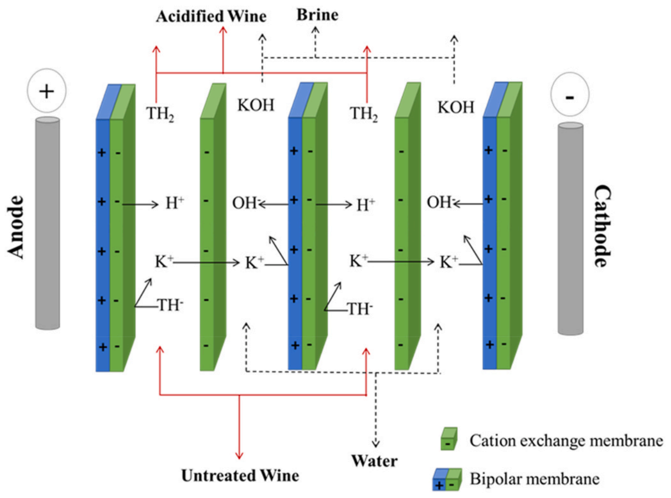

Fig. 7. Acidification process by bipolar electrodialysis (Adapted from El Rayess & Mietton-Peuchot, 2016).

- Improved sustainability: wine acidification by bipolar ED can contribute to improved sustainability in winemaking. Reducing the need for excessive additives, it minimizes waste and resource consumption. Also, the effluent recovery (KOH) can be used as a cleaning solution. Each litre of treated wine consumes less than 0.002 kW making this technique a low energy consuming.

Musts and wines can sometimes present excessive acidity depending on the climate, region, season and grapevine variety. In cold-climate wine regions, grape berries are rich in acidity as the low temperature favours the acid respiration process (Cioch-Skoneczny et al., 2021). This excessive acidity may lead to unbalance wines with organoleptic defaults as a “sour” taste and to difficulties in the fermentation process especially if the pH is below 3.0.

Deacidification, which is the process of reducing titratable acidity in musts and wines, is the solution for the excessive acidity issue. Many strategies can be implemented for the deacidification like the microbiological degradation of malic acid, the precipitation of potassium bitartrate, the chemical deacidification with neutral potassium tartrate, calcium carbonate and potassium hydrogen carbonate, or blending with musts and wines with low acidity (Comuzzo & Battistutta, 2019).

Similar to acidification, the bipolar ED can be an alternative solution for wine deacidification. The process is similar to the acidification one, but the cation exchange membranes are replaced by the anion exchange membranes to extract the excess of tartaric acid and malic acid while keeping the potassium in the wine. The application of the electric current will move the anions (TH^- and M^{--}) towards the anode. They will cross the anion exchange membrane but they are stopped by the bipolar membrane. These anions will be found in the water compartment and will be eliminated. On the contrary, potassium ions will tend to move to the cathode but will be stopped directly by the bipolar membrane. Also, due to the dissociation of water in the junction layer of the bipolar membrane, the wine will be enriched by the OH^- anions (El Rayess & Mietton-Peuchot, 2021). All these factors will result in the decrease of the titratable acidity and the increase of the pH. This technique was accepted by the OIV in 2012 and by the European Community in 2013 (Regulation EU 144, 2013).

4. Partial and total dealcoholization of wine

Due to new consumption habits, several researches proved that wine has evolved from a meal component to a cultural beverage. Contextual factors, such as social interaction, place and moment of consumption, are known to significantly impact product consumption and evaluation for low-alcohol wine or completely dealcoholized wines (Stasi et al., 2014). Consequently, the development of new products with low or no alcohol content could be very interesting to answer to these new markets. Over the last two decades, an increase in alcohol content in wine has been observed. Various causes have led to this rise in alcohol content in wine, including global warming and the quality policy implemented by professionals in the wine industry. These factors and practices led to high sugar concentrations in the grapes resulting in unbalanced wines. In addition, alcohol consumption has grabbed the attention of governments. In recent years, more restrictive rules on alcohol consumption and responsibility have been enforced. Awareness campaigns have focused on the message of reducing alcohol consumption for social and health reasons.

For all these reasons, research has been developed to test different strategies and techniques to decrease the alcohol content in wines. Depending on the moment of application, these strategies can be divided into: i) pre-fermentation dealcoholization; ii) during fermentation; iii) post-fermentation dealcoholization.

4.1. Reduction of must sugars (pre-fermentation dealcoholization)

One of the strategies applied to reduce alcohol content is to decrease the sugar content in musts. This strategy using membrane techniques was forbidden in Europe until 2012 when a specific application on the reduction of must sugar content through membrane coupling (UF and NF) was adopted (Resolution OIV-OENO 450B-2012, International Organisation of Vine and Wine, 2012). It was patented by Bucher Vaslin company and marketed under the name of REDUX®. It consists of must filtration through UF as the first step. The resulting permeate is composed of water, sugars, acids, minerals and some anthocyanins and tannins. This permeate is then treated by NF resulting in a viscous retentate, rich in sugars. The permeate of NF is mainly composed of water, acids and minerals, while the retentate of UF is formed by polysaccharides, proteins, anthocyanins and tannins, which are reincorporated into the must that is being treated. Wines made from reconstituted and treated must present lower alcohol content. To reduce the alcohol content by 1–2% v/v, a volume of 20–25% of grape must should be treated by REDUX® process (El Rayess & Mietton-Peuchot, 2021). This process is preferred over the one-step treatment of NF because UF treatment leads to lower osmotic pressure and macromolecules content resulting in higher sugar concentration by the NF process. The higher sugar concentration allows the decrease in volume loss during treatment. Salgado et al. (2015) demonstrated that two-stage NF process yielded better results than one-step NF for sugar reduction in grape musts.

The strategy to reduce sugars before fermentation by membrane coupling has significant advantages on winemaking process, especially on the fermentation and wine quality. Reduction of sugars before fermentation reduces the osmotic stress at the beginning of fermentation and the toxic stress due to elevated alcohol at the end of fermentation. Furthermore, this process tends to preserve the organoleptic quality of the resulting wine, as the aromas formed during fermentation and aging are not affected by this process.

4.2. Wine alcohol removal (post-fermentation)

Another strategy to reduce alcohol content in wines is to remove ethanol in post-fermentation. Herein, membrane processes, such as RO, NF, osmotic distillation (OD) and pervaporation (PV), use different membrane materials and mechanisms to achieve the removal of ethanol while retaining essential wine components.

4.2.1. Nanofiltration and reverse osmosis

NF and RO constitute promising alternatives to thermal-based processes for wine dealcoholization because they remove alcohol under mild temperature conditions preserving the quality of wine and the bioactive compounds. In 1975, German brewing company Lowenbrau obtained the first patent for the application of RO for dealcoholization of beer and wine (El Rayess & Mietton-Peuchot, 2016). During RO treatment, water and ethanol pass through the dense membrane with a low molecular weight cut-off (MWCO) when the pressure difference across the membrane is higher than the osmotic pressure difference. RO is coupled to a column distillation in order to limit the volume loss by recirculating water resulting from distillation into the original wine.

Gonçalves and de Pinho (2003) proposed a patent (WO 2004/113, 489) for alcohol removal from wines based on the use of NF membranes coupled to a distillation process. The NF process was preferred to RO because it provides higher alcohol flow rates together with greater permeation rates. Subsequently, Pilipovik and Riverol (2005) investigated the RO for the total dealcoholization of beverages and found that RO is not economically viable for producing low-alcohol beverages. Labanda et al. (2009) compared NF and RO membranes for wine dealcoholization. They found that both processes allow ethanol rejection up to 36%. In their study, Catarino and Mendes (2011) conducted experiments involving various RO and NF membranes to produce wine with reduced alcohol content. Their findings revealed that RO membranes exhibited the lowest ethanol rejection but also the lowest permeate fluxes. Conversely, the tested NF membranes, except one, demonstrated high permeate fluxes, effective ethanol rejection, and efficient removal of aroma compounds. Additionally, a notable advantage of employing NF membranes was the enrichment of ethanol in the permeate compared to that obtained with RO membranes.

Gil et al. (2013) investigated the use of RO for partial dealcoholization of two red wines. It was found that partially dealcoholized wines (−1 and −2 vol. %) were very similar in their composition to the control wines. During this study, RO is deemed highly valuable for the partial dealcoholization of red wines as it minimally affects their composition and sensory attributes. On the contrary, Meillon et al. (2009) demonstrated that partial dealcoholization of Merlot and Syrah wines can affect sensory perception. In different studies, RO was tested in combination with OD for wine dealcoholization (Pham et al., 2020; Russo et al., 2019). Both studies showed that dealcoholization using combined technologies did not significantly affect wine aromas and flavor.

4.2.2. *Osmotic distillation*

OD is a non-pressure-driven membrane process used for the extraction of water and other solvents from solutions under atmospheric pressure and room temperature (Cassano et al., 2020). In this process, a membrane contactor technique is employed using hydrophobic polymeric membranes, such as PP, PVDF, polyethylene and polytetrafluoroethylene, due to their low surface tension values. During wine dealcoholization, the microporous membrane (0.2 μm) serves as a vapor barrier separating two aqueous solutions, namely the feed (wine) and the stripper agent (water) where the solutions are flowing a countercurrent direction. This membrane facilitates the mass transfer between these solutions without causing any mixing or dispersion of one phase into the other. As a result, it selectively removes ethanol from the wine while preserving its other components. The mechanism of ethanol transport in the process of wine dealcoholization through membrane contactor can be segmented into three distinct steps:

i) evaporation of ethanol occurring at the membrane pores on the wine side;
ii) diffusion of ethanol vapor through membrane pores;
iii) condensation of ethanol vapor in the stripping solution.

The first introduction of OD for wine dealcoholization was realized by Hogan et al. (1998). Their key findings revealed that by subjecting high-alcohol wines to OD, operating at temperatures ranging between 10 and 20 °C and employing water as the stripping agent, it was possible to rapidly achieve a significant reduction in alcohol content up to 6%.

The impact of the type of stripping solution and the operating conditions (feed and stripping velocities, temperature) on ethanol removal during wine dealcoholization using OD was investigated by Varavuth et al. (2009). The most promising result was obtained when using water as stripping allowing the reduction of ethanol content up to 34%. Increasing the feed and stripping solution velocities and system temperatures (from 25 to 45 °C) enhanced the ethanol flux and thus ethanol removal performance; unfortunately, a significant loss of aroma compounds was reported due to its undesired extraction along with the ethanol. This latter point is credited to the high affinity of most organic compounds toward ethanol, which may result in substantial changes in the organoleptic features of the final wine. More recently, Liguori et al. (2013a) found that the optimal ethanol removal from model solutions was obtained when working in laminar conditions for both feed and stripping streams. They also noted a reduction in ethanol transport rate as the ethanol content of the solutions increased, which they attributed to saturation effects. Furthermore, they found that increasing temperatures had an accelerating effect on the dealcoholization process.

Lisanti et al. (2013) studied the impact of partial dealcoholization of two red wines by OD at three levels (−2, −3 and −5 %v/v) on the quality of dealcoholized wines. The results demonstrated that both −2% wines were not perceived as different from the standard wines while both −5% wines were different. Dealcoholized wines showed modified sensory profiles with a notable decrease in “Red fruits”, “Cherry” and “Spicy” notes. The effect of total dealcoholization of red wine by OD was evaluated by Liguori et al. (2013b). No significant differences were found in terms of pH, total acidity, composition of organic acids, total phenolics and flavonoids. However, color intensity and tonality were significantly affected. The volatile compounds were severely impacted with a decrease over 98%. The authors explored also the impact of partial and total dealcoholization of white wine by OD on the chemical composition and volatile compounds content (Liguori et al., 2019). No significant changes in total acidity, organic acids, total phenols and flavonols were detected during the dealcoholization process. On the contrary, the volatile compounds decreased with ethanol removal reaching an overall loss of 96% in totally dealcoholized wines. For instance, alcohols, esters and lactones were the most impacted volatile compounds. Corona et al. (2019) explored the impact of various degrees of wine dealcoholization through OD on the volatile compounds, phenols and sensory attributes of red wine (13.2 %v/v). The reduction in alcohol content resulted in the following alcohol levels: −4.9%, −6.3%, −7.8%, −9.2% and −10.5% (v/v). The study findings revealed that wines with a decreased alcohol content of 4.9% and 6.3% exhibited satisfactory retention of esters (84% and 82% respectively). The color and taste of all the dealcoholized wine samples remained unaltered.

4.2.3. *Forward osmosis in wine dealcoholization*

Forward osmosis (FO), an emerging membrane technology, is still in development for several applications such as water desalination (Castro-Muñoz, 2023), brine concentration, wastewater treatment (Vital et al., 2018), among others. In food science, this latter technology has also been used in the concentration processing of grape juice (Kim et al., 2019; Tavares et al., 2022) and dealcoholization of alcoholic beverages. The concentration of complex extracts (such as juice) is achieved by osmotic dewatering; unfortunately, FO still lacks in long-term stability in this application, as the flux decreases due to the severe fouling (Blandin et al., 2020; Kim et al., 2019). Despite this drawback, resulting juices concentrated via FO present high-quality retaining all the initial nutrients. Considering the dealcoholization, FO has been mainly involved in the extraction of minor compounds (such as ethanol) diluted in aqueous systems. For instance, beer dealcoholization (Ambrosi et al., 2020) and kiwi wine dealcoholization (Huang et al., 2022) are relevant cases of study at lab-scale in this field. Using $MgCl_2$ as an ethanol extracting solution in FO, the final wine samples contained ca.0.45 v/v ethanol content while presenting improved phenolic (up to 91.6 μg/mL) and volatile organic compounds content (Huang et al., 2022).

4.2.4. *Wine dealcoholization via membrane contactors*

Membrane contactors (MC) have also been applied for the partial dealcoholization of wine. Compared with FO, MC technology has been deeper in wine processing. In this technology, hydrophobic MC membranes along with aqueous stripping solutions in many cases are applied (Lee et al., 2023). For instance, Diban et al. (2008) demonstrated the viability of MC pilot plant applications for partial dealcoholization of wine using a commercial PP hollow fiber membrane contactor (Liqui-Cel® Extra-Flow 2.5 in. × 8 in., 1.4 m^2, Celgard). This approach was successfully experimented with an alcohol reduction of 2% (v/v) in Merlot Grape variety wine containing 10–13% (v/v); however, the authors noted a major loss of volatile aromas compared to ethanol. Also, hydrophobic aroma compounds, such as ethyl octanoate, exhibited an adsorption phenomenon on the hydrophobic membrane representing a 2–3% aroma loss. In a more recent study, Gambuti et al. (2011) used a similar commercial MC module for the partial removal of ethanol from red wines. Herein, partial dealcoholization of 2, 3 and 5% v/v were obtained for Aglianico, Merlot and Piedirosso wines, respectively, while maintaining the content of phenolic compounds and natural coloring substances of red wine. Interestingly, the authors observed the loss of monomeric anthocyanins attributed to their adsorption on membrane surface or oxidation when in contact with air. To minimize such losses, it is suggested that the dealcoholization treatment could be performed using wines containing adequate content of SO_2 under conditions limiting the O_2 dissolution.

Even if partial wine dealcoholization has been successfully done via MC, it is worth mentioning that using water as a stripping agent may alter to some extent the oxygen isotopic ratio of the wine, as declared by Ferrarini et al. (2016). Therefore, this latter effect could be confused with dewatering due to the net water transfer from the stripping side toward the wine side.

4.2.5. *Wine dealcoholization via pervaporation*

As one of the most selective membrane technologies, pervaporation (PV) stands out as a potential technology for the dealcoholization of wine. In principle, the removal of ethanol from aqueous systems (like low ethanol content compared to water in wine system) requires organophilic membranes, which may display facilitated transport of ethanol molecules while hindering water molecules. Compared to other dealcoholization techniques such as traditional distillation, PV demands less energy expenditure, is highly selective (i.e., high ethanol separation efficiency) and easy to operate, and selectively removes the minor component (Castro-Muñoz, 2019).

Using this technology, Tan et al. (2003) applied a polydimethylsiloxane (PDMS) membrane displaying a permeate rate as high as 1.5 kg/m^2 h when operating at 40 °C. To some extent, the wine contained initially 11.5 % ethanol, which exhibited between 3 and 7 % after PV dealcoholization. In subsequent work, the application of a commercial membrane organophilic PERVAP Sulzer 1060 resulted in a successful dealcoholization of typical Hungarian wine (known as Tokaji Harslevelu). Interestingly, when the operating temperature increased from 40 to 70 °C, the permeation was also increased from 0.287 to 1.2 kg/m^2 h, respectively, but worsened separation efficiency (Takacs et al., 2007). Here, the authors also noticed an ethanol permeation increase as a function of temperature; however, it was pointed out that lower temperature is suggested for better preservation of aroma compounds, which consequently affect the organoleptic properties of the product. This latter point came out after observing that the obtained permeate samples presented high ethanol concentrations (35–38 %) with the presence of aroma compounds. This is a common issue in wine alcoholization since most of the fragrances, aroma and flavoring compounds in wine present a high affinity to ethanol, serving as carrier of such compounds out from the main product. To avoid the loss of aromatic compounds, Catarino and Mendes (2011) included PV for the aroma compounds extraction before the dealcoholization using NF and RO stages. In this, study, organophilic polyoctylmethylsiloxane (POM)--based membranes have been used to restore mostly esters in the dealcoholized wine. Similarly, Salgado et al. (2017) employed PV for the initial extraction of aroma compounds from the must for a subsequent blending with dealcoholized wine, as illustrated in Fig. 8. In this complete dealcoholization process, PV has been strategically used to extract the aroma compounds from the must. After ethanol removal and reduction of sugar content using NF stages, the dealcoholized wine containing approximately 10% ethanol was enriched with aroma fractions. To some extent, several aroma compounds, such as isoamyl alcohol, hexanal, 2-phenylethanol and benzaldehyde, were recovered thanks to the great organophilic properties of the POM membrane.

So far, PV has been mainly involved in removing ethanol fractions from wine or recovering aromas for a further enriching low-alcohol content wine; however, Coronas' research group recently proposed PV for extracting water from dealcoholized wine via osmotic distillation. This extracting water (containing up to 5.3 wt% ethanol) was pervaporated with hydrophobic (PDMS or zeolite silicalite-1) and hydrophilic (zeolites mordenite or faujasite) membranes (Esteras-Saz et al., 2023). Due to the usage of such membranes, two main products were obtained, such as bioethanol (recovering 88% of the ethanol removed from the wine) and a 99.4 wt% water product. This latter product presented suitable physicochemical properties to be reused as extracting water in the subsequent osmotic distillation process of wine.

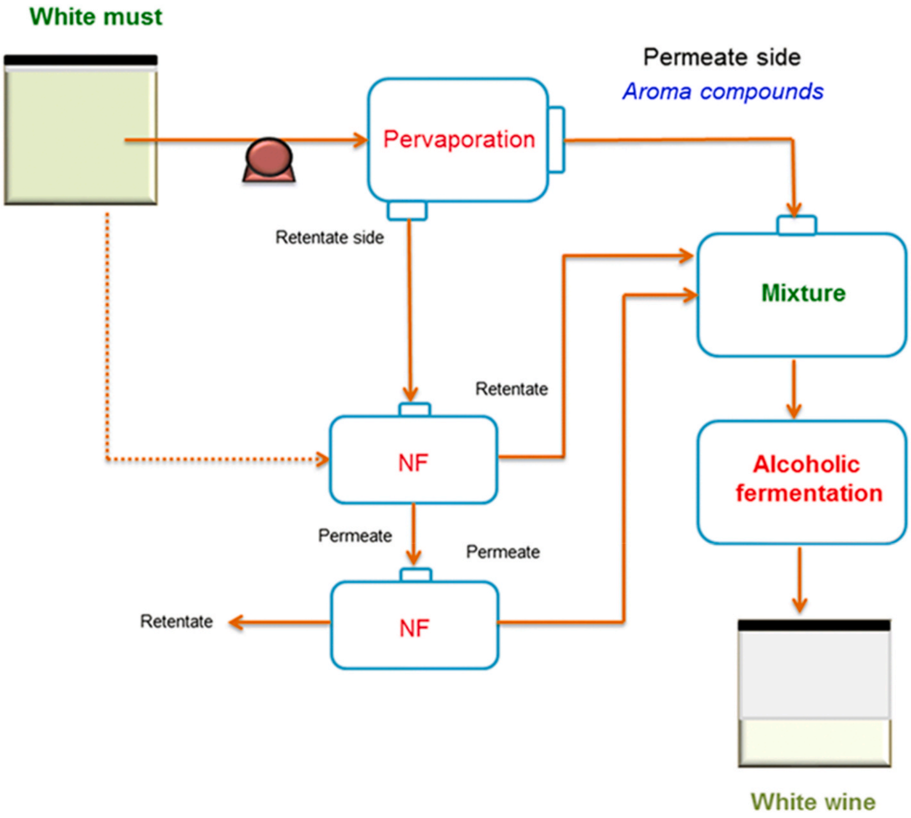

Fig. 8. PV-assisted dealcoholization process of wine. Adapted from Salgado et al. (2017).

5. Conclusions and recommendations

Membrane-based processes are well-established technologies in the processing of several beverages, including wine and must, thanks to their advantages over conventional separation methodologies, such as no use of chemical reagents and additives, low processing temperatures, good selectivity, no phase change, strong adaptability, easy scale-up and scale-down, among others. In this review, the impact of membrane-based processes on the winemaking industry has been reviewed in the light of the recent studies concerning both conventional and emerging membrane technologies.

MF is commonly used in wine production to remove suspended solids and turbidity from both wine and must. Outstanding progress has been reported in the literature concerning the development of low-fouling materials, identification of wine molecules responsible for membrane fouling and methods to mitigate such phenomenon. Other membrane applications in wine-making processes have been extensively studied in recent years. Among them, ED and bipolar ED offer very attractive solutions for tartrate stabilization, acidification and deacidification of wines. In this field, new progress in membrane synthesis and manufacturing provides a good contribution to limiting and controlling different aging mechanisms including fouling phenomena.

Different membrane processes, including NF, RO, OD, FO, MC and PV represent useful post-fermentation approaches to produce low-alcoholic wines limiting the degradation of bioactive compounds (e.g., polyphenols, antioxidants, etc.). All these processes, including integrated systems, can be useful tools to redesign the traditional process of wine manufacturing within the logic of the process intensification strategy with significant advantages in terms of energy saving, reduction of produced waste and environmental impact, improved quality and preservation of the intrinsic organoleptic characteristics of original grape varieties.

Despite all the progress and studies realized in the field of membrane process applied to wine industry, further research needs to be done such as:

- The elucidation of wine physico-chemical interactions between wine molecules themselves and with the membrane to better understand fouling in driven-pressure processes.
- The exploration of the impact of new membrane materials on processes performances and the wine quality.
- The study of fouling during wine treatment by electrodialysis.
- Better understanding of the dynamic filtration for wine and by-products clarification.
- The study of the effect of operating conditions on fouling during gas transfer management by membrane contactors.
- Environmental life cycle assessment of membrane processes used in wine industry.
- In wine dealcoholization, wine's quality and organoleptic properties need to be monitored, as the high affinity of the organics (aromas, antioxidants) in ethanol may cause a loss and strong change in the organoleptic features of the final products. For instance, when using membrane processes with dense membranes (e.g., PV), the dealcoholization process will be safe for preserving non-volatile compounds from the wine as their permeation will be complicated. In these processes, researchers need to focus on the membrane type (hydrophilic and hydrophobic/organophilic) to some extent limit the permeation of volatile aroma substances when removing ethanol. On the other hand, when using membrane processes with porous membranes (e.g., FO, MC), more attention should be paid to the permeation of both non-volatile and volatile compounds contained in the wine, especially in MC processes that have demonstrated very low selectivity toward low molecular weight solutes and gas state species (like volatile compounds) (Simons et al., 2009).

Credit authors statement

Youssef El Rayess: Conceptualization, Visualization, Methodology, Writing-original draft; Roberto Castro-Muñoz: Conceptualization, Visualization, Writing-original draft; Alfredo Cassano: Conceptualization, Visualization, Writing-original draft, Writing-review & editing.

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.

Data availability

Data will be made available on request.

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Abbreviations

ED	electrodialysis
FO	forward osmosis
MC	membrane contactor
MF	microfiltration
MWCO	molecular weight cut-off
NF	nanofiltration
PES	polyethersulfone
PP	polypropylene
PS	polysulfone
PV	pervaporation
PVDF	polyvinylidene fluoride
RO	reverse osmosis
RVF	rotating and vibrating filtration
TMP	transmembrane pressure
UF	ultrafiltration

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