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Evaluating membrane behavior to ethanol-water mixtures and wine: A comparative investigation

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ABSTRACT

In this study the performance of three nanofiltration membranes (TS 40, NF99, HL) and one reverse osmosis membrane (RO-SE) while filtering ethanol-water mixtures (0–10.5% v/v) and a white wine (10.5% v/v) was evaluated. The experiments were conducted using water, ethanol-water mixtures, and white wine at varying pressure (0–20 bar, 21 ± 1 °C) to explore the impact of pressure on permeate flux and permeability. Further tests were performed with white wine and ethanol-water mixture (10.5% v/v) at pressure 20 bar and 21 ± 1 °C up to volume reduction factor of 4 to evaluate performance (based on permeate flux, permeability, fouling index, ethanol rejection and retention of selected compounds) of different membrane. Among the investigated membranes the HL membrane exhibited the highest permeate flux consistently across varying operational pressures, showcasing superior permeability. HL and NF99 membranes showed greater effectiveness in reducing the alcohol content in wine, with ethanol rejection rates of 5.14% and 5.46%, respectively. Conversely, RO-SE (10.64%) and TS 40 (18.30%) exhibited the highest ethanol rejection rate. The fouling index for all the membranes ranged between 22.5 and 43.5%. In addition to this NF and HL also showed highest rejection towards reducing sugars (>90%), glucose (>80%), fructose (>88%), citric acid (>88%) and tartaric acid (>89%) in dealcoholized wine. Overall, HL and NF99 membranes appear to be the most effective options for wine dealcoholization.

1. Introduction

The consumer demand for low, reduced, and dealcoholized wine is increasing in recent years in many countries of the world and more and more producers are investing in this segment in the light of increasingly "healthy" consumption (Bucher et al., 2020; Research Reports World, 2023; Valentepali, 2023). The global non-alcoholic wine market size was valued at USD 1469.15 million in 2022 and it is expected to reach USD 4546.76 million in 2028, with a compound annual growth rate (CAGR) of 20.72% during 2022–2028 (Global Non-Alcoholic Wine Market Research Report, 2023). The primary markets were France at USD 178 million, Germany at USD 74 million, Italy at USD 32.7 million, and Spain at USD 16 million (Valentepali, 2023). In the world, 50% of the adult population does not consume alcoholic beverages, citing reasons like religious beliefs, health concerns, personal taste, or a shift towards non-alcoholic alternatives (Sam et al., 2021; Valentepali, 2023).

The removal of alcohol in wine can be achieved intervening in three different stages of the wine production: (i) before wine fermentation through the removal of fermentable sugars; (ii) during fermentation through the reduction of the ethanol production; (iii) after fermentation through the separation of alcohol by thermal distillation or membrane operations (Ozturk & Anli, 2014; Sam et al., 2021). Alcohol content can be reduced by up to 2–7% v/v through techniques employed during the pre-fermentation and fermentation stages, resulting in what is characterized as partially dealcoholized wine (Kumar et al., 2024). However, wines produced using such techniques often exhibit poor sensory qualities and show significant limitations when applied on an industrial scale. On the other hand, dealcoholized or alcohol-free wines (with alcohol content below 0.5% v/v) are exclusively produced by employing post-fermentation techniques on finished wines. Final dealcoholized wines appear to be much more versatile, reliable, and suitable for scale-up at an industrial level (Pickering, 2000). Low-temperature

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distillation and spinning cone columns are well-established techniques used for the removal of ethanol from wines (Osorio Alises et al., 2023; Pham et al., 2019; Puglisi et al., 2022). However, these techniques lead to significant changes in the concentration of wine constituents (volatile and non-volatiles) and sensory characteristics, depending on the extent or quantity of ethanol that is removed. Liguori et al. (2019) processed Falanghina white wine to dealcoholization, reducing its alcohol content from 12.5% to 0.3% v/v using osmotic distillation, and observed significant reductions in various volatile compounds, including total higher alcohols (99%), total esters (99%), total acids (98%), and total ketones and lactones (99%). Furthermore, the dealcoholized wine exhibited a decrease in overall acceptability, appearance, odor, aftertaste, body, and sweetness, while perceptions of acidity increased compared to the original wine. On the other hand, Ju et al. (2023) produced a Muscat white with 6% v/v ethanol using distillation. The distilled wine exhibited notable reductions in various volatile compounds, including total alcohols (−7%), total esters (−24%), total aldehydes (−16%), and total terpenoids (−42%). In terms of sensory characteristics, the wine showed a decrease in aroma intensity, aroma purity, and wine body, with no significant change in aftertaste.

Membrane filtration techniques - including microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) - have long been utilized within the wine industry for different applications such as must and wine clarification, cleaning, stabilization and sterilization (Banvolgyi et al., 2006; García-Martín et al., 2009; Pati et al., 2014). These processes offer particular advantages over conventional separation technologies (distillation/multistage distillation, extraction, etc.) in terms of low energy, low cost, mild temperatures, high efficiency and no-additives requirement (Guiga & Lameloise, 2019); i.e. during osmotic distillation of wine different types of stripping solutions, such pure water, 50% (w/w) glycerol, and 40% (w/w) $CaCl_2$ were used (Varavuth et al., 2009).

Among pressure-driven membrane operations, NF and RO can be used directly in the treatment of finished wine to produce low-alcohol wines (Mangindaan et al., 2018; Massot et al., 2008; Russo et al., 2019; Schmidtke et al., 2012). These processes are based on the use of composite membranes with a high strength polymer as a supporting layer which allow the permeation of water, ethanol and low molecular weight compounds (i.e., acetic acid, lactic acid) while retaining larger molecules such as sugars, higher acids, higher alcohols and phenolic compounds (Banvolgyi et al., 2016). Furthermore, utilizing NF and RO membranes for dealcoholization at low temperatures helps maintain the organoleptic properties of the original wine (Ozturk & Anli, 2014).

For every membrane process, the selection of a suitable membrane is essential to achieve an optimal separation (Verhoef et al., 2008). The selectivity and performance of a membrane are contingent upon various factors, including thickness, porosity, the structure of its top layer (porous or dense) (Peng et al., 2010, 2021), geometric configuration, porosity (Madaeni, 2001; Muller et al., 2020), and material properties like glass transition temperature, composition, hydrophobicity/hydrophilicity and surface charge (Sam et al., 2021; Thai et al., 2021; Verhoef et al., 2008). NF membranes exhibit superior permeate fluxes compared to RO membranes and provide better rejection for smaller molecules (such as sugars, peptides, proteins, etc.) at about 75 bar than ultrafiltration (UF) membranes (Massot et al., 2008; Sam et al., 2021). RO membranes operates at a slower pace than NF and is less cost-effective in this context (Yadav et al., 2022).

The performance of different membrane has been analyzed in various fields of within the industry such as concentration of fruit and vegetable juices (Gaglianò et al., 2022; Mondal et al., 2021), the recovery of polyphenolic compounds and aromas, fragrances and essential (AFE), biologically active compounds (Arboleda Mejia et al., 2020; Castro-Muñoz et al., 2023; Conidi et al., 2022), wastewater treatment, the production of sparkling water (Ahmad et al., 2021; Moradihamedani, 2022), and in the production of dealcoholized beers (Bóna et al., 2023; Varga et al., 2023). As well as, in recent years, various types of membranes have been investigated for their application in the dealcoholization of wine by numerous researchers (Banvolgyi et al., 2006, 2016; Catarino & Mendes, 2011; Ivić et al., 2021a; Ivić et al., 2021b; Labanda et al., 2009; Salgado et al., 2017). However, the majority of studies have focused on red wines and the use of specific membranes. Therefore, further research is required to explore the dealcoholization of white wines encompassing various grape varieties, as well as the utilization of different membranes with their respective specifications, allowing for comparative analyses against other membranes.

In this context, the present study was aimed at comparing the performance of three different NF membranes (TS 40, NF99, HL) and one RO membrane (RO-SE) for the dealcoholization of both ethanol-water mixtures and white wine in selected operating conditions. In particular, the investigation focused on the evaluation of the membrane productivity under different transmembrane pressure conditions as well as of the ethanol removal in appropriate operating conditions of pressure, temperature and volume reduction factor (VRF). The investigated membranes were also compared for their performance in reducing the alcohol content of wine (initially 10.5% v/v) in a diafiltration mode. This approach, based on the addition of water to the retentate, improves the ethanol removal selectivity allowing to keep constant the concentration of non-permeable species as well as of the osmotic pressure so minimizing concentration polarization and permeate flux reduction. The impact of both NF and RO processes on physico-chemical parameters of dealcoholized wine was also assessed.

2. Materials and methods

2.1. Ethanol-water solutions and wine

Ethanol-water solutions were prepared by mixing ethanol (99%, VWR chemicals) and distilled water, at different ethanol content (0.5, 3, 7 and 10.5% v/v). The selected concentrations were chosen based on proposed wine categories by Pickering (2000) and Saliba et al. (2013), which are delineated by their alcohol content as: alcoholic (>10.5% v/v ethanol), lower-alcohol (5.5–10.5% v/v ethanol), reduced-alcohol (1.2–5.5% or 6.5% v/v ethanol), low-alcohol (0.5–1.2% v/v ethanol), and alcohol-free (0.5% v/v ethanol) wine. Commercial white wine (Sancrispino, Cantine Ronco, Forlì (FC), Italy) - a blend of Trebbiano, Pinot, Chardonnay and Malvasia Italian grapes - with an alcohol content of 10.5% (v/v), was purchased from a local supermarket. The choice of white wine was made considering that white wines are expected to be closer in composition to the hydro-alcoholic solutions used as references, presenting a lower load of compounds other than ethanol. Therefore, in this investigation, the membrane filtration experiments were performed on white wine only.

2.2. Membranes

In this study, one RO and three NF membranes, all in flat-sheet configuration, were used. Their detailed characteristics are presented in Table 1. The selection of membranes in this study was based on their commercial availability, which would allow for easy scale-up to the industrial level. Specifically, three nanofiltration (NF) membranes were chosen because they have higher flux rates, which can help speed up the overall process, as different NF membranes can have varying characteristics that may impact the process. In contrast, only one reverse osmosis (RO) membrane was used for comparison purposes.

2.3. Experimental setup and procedures

Alcohol removal experiments from hydroalcoholic solutions and white wine were performed in a dead-end stirred filtration cell (Sterlitech™ HP 4750, Kent, WA, USA) with an effective membrane filtration area of 13.85 cm^2 and a maximum volume capacity of 300 mL. A nitrogen cylinder, equipped with a two-stage pressure regulator, was

Table 1
Characteristics of selected membranes for dealcoholization.

Membrane type	Nanofiltration			Reverse osmosis
	NF99	TS40	HL	RO-SE
Supplier	Alfa-Laval	Trisep™	GE Water & Process Technologies	GE Water & Process Technologies
Configuration	flat-sheet	flat-sheet	flat-sheet	flat-sheet
Membrane material	TFC composite	Piperazine polyamide	Piperazine polyamide	Polyamide
Nominal MWCO (Da)	200	200–300	150–300	<100
$MgSO_4$ rejection (%)	>98	99	98	–
NaCl rejection (%)	–	40–60	–	99
pH operating range	3–10	1–12	3–9	1–11
Max. operating temperature (°C)	50	50	50	50
Max. operating pressure (bar)	55	41	–	–
Contact angle (°)	35[a]	34[b]	38[b]	–
Water permeability at 21 ± 1 °C (L/m^2hbar)	7.52	10.14	11.08	1.45

MWCO, molecular weight cut-off; data from.
[a] Ruiz-Gutierrez et al. (2024).
[b] Żyłła et al. (2021).

connected to the top of the stirred cell to supply the desired pressure for filtration experiments. Stirring inside the cell was accomplished by using a magnetic stirrer. The experimental setup is illustrated in Fig. 1.

A first set of experiments was carried out by using three different feed solutions: water, ethanol-water mixtures of different concentration (starting from the lowest, 0.5–10.5% v/v) and white wine. These experiments were performed at different operating pressures (ranged from 0 to 20 bar) and at a consistent temperature of 21 ± 1 °C, according to a batch concentration configuration, in order to study the effect of pressure on the permeate flux. The permeate flowrate was determined using a graduate cylinder and a stopwatch.

Additional experiments were performed with wine and 10.5% ethanol/water mixtures (HS), at an applied pressure of 20 bar and a temperature of 21 ± 1 °C. For each experiment 200 mL of feed solution were used and the permeate was removed continuously from the membrane cell, according to a batch concentration mode, up to collect a permeate volume of 150 mL, corresponding to a volume reduction factor (VRF) of 4.

VRF was estimated according to equation (1) (Destani et al., 2020):

$$VRF = \frac{V_f}{V_r} \quad (1)$$

where V_f is the initial feed volume (mL) and V_r is the retentate volume (mL).

In order to improve the removal of ethanol, diafiltration experiments were performed by adding distilled water to the retentate according to a batch diafiltration process (also called discontinuous diafiltration). Specifically, after a concentration factor of 4 was achieved, the retentate was diluted by adding the same volume of distilled water (50 ml). Four discontinuous diafiltration (adding a total volume of 200 mL of distilled water to the retentate) were performed for the NF membranes before stopping filtration. On the other hand, two discontinuous diafiltration (adding a total volume of 100 mL of distilled water to the retentate) were performed for the RO-SE membrane. This approach ensured the stability and concentration of non-permeable compounds throughout the diafiltration cycles.

After each experiment, the membrane's water permeability was assessed. Then membranes were submitted to a standard cleaning-in-place protocol consisting of a water cleaning for 10 min followed by an alkaline cleaning with a commercial detergent (Ultrasil WA 0.2% v/v, 40 °C, 60 min) and a final rinsing with distilled water. This cleaning process restored the original permeability of the membrane before starting further experiments with the same membrane. The permeate flux and permeability were calculated using Equations (2) and (3), respectively (Destani et al., 2020; Ivić et al., 2021a).

$$J = V/(A \times t) \quad (2)$$

$$L_p = J/P \quad (3)$$

where J (L/m^2h) is the permeate flux; L_p (L/m^2h bar) is the permeability; P (bar) is the applied pressure; V (L) is the collected permeate volume during the process; A (m^2) is the effective membrane area; t (h) is the operating time.

The fouling index (FI) of NF and RO membranes can be determined using different approaches (Choi et al., 2009). In this study it was determined by measuring the hydraulic permeability of the membranes by using the following equation (Ivić et al., 2021a):

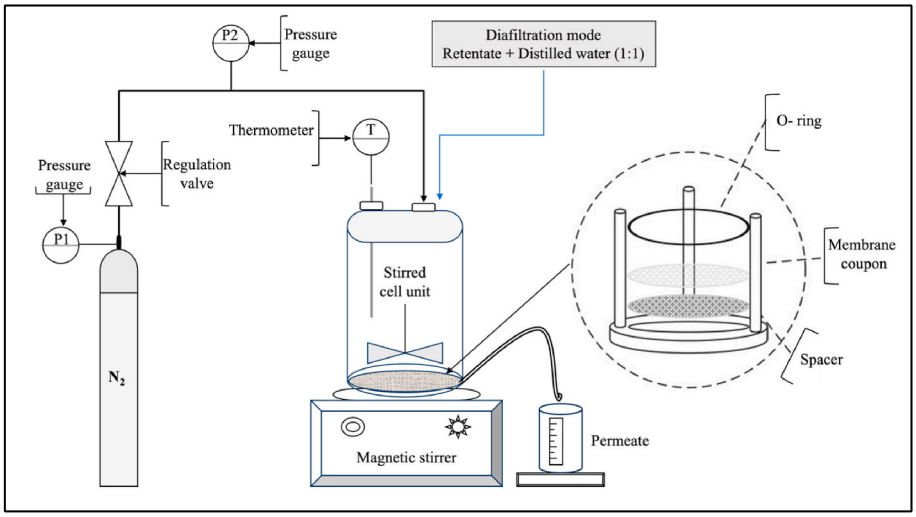

Fig. 1. Scheme of the experimental set-up of membrane filtration for dealcoholization process.

$$FI(\%) = \left(1 - \frac{L_{p1}}{L_{p0}}\right) * 100 \quad (4)$$

where L_{p1} and L_{p0} are the pure water permeability (L/m^{2}hbar) before and after wine concentration, respectively.

2.4. Oenological parameters analysis

The basic oenological parameters, including ethanol, pH, total acidity, volatile acidity, citric acid, glucose, fructose, and residual sugar of both retentate and permeate after the process, were measured by FTIR spectroscopy using the BACCHUS 3 multispec model (TDI, Barcelona, Spain). Total Soluble Solids (TSS) measurements were carried out at 20 °C by using a hand refractometer (Atago Co., Ltd., Tokyo, Japan) with scale range of 0–32°Brix. The color parameters, including absorbance at 420, 520 and 620 nm, intensity (420+520+620 nm), tone (420/520 nm), chroma, hue, as well as L^* (perceptual lightness), a^* (red/green value), and b^* (yellow/blue value) values were measured using a CIELab instrument (Smart analysis DNA phone Srl, Parma, Italy).

The overall rejection (R) of selected membranes towards various components was calculated by using Equation (5) (Banvolgyi et al., 2016).

$$R(\%) = \left(1 - \frac{Cp,i}{Cr,i}\right) * 100 \quad (5)$$

where, $C_{p,i}$ represents the compound i concentration in the permeate, while $C_{r,i}$ stands for its concentration in the retentate side.

2.5. Statistical analysis

All the experiments were performed in triplicate, and results were reported as averages ± standard deviations (SD). Linear regression analysis was done to obtain the regression equation and coefficient of determination (R^2) for all parameters. Tukey test ($p < 0.05$) was carried out to compare means in the ANOVA using Minitab Statistical software v.18 (State College, PA, USA).

3. Results and discussion

3.1. Impact of hydroalcoholic solutions and wine on transmembrane flux and permeability

Fig. 2 illustrates the trend of transmembrane flux for each selected membrane for distilled water, hydroalcoholic solutions at different concentrations and wine under varying operating pressure conditions and at a consistent temperature of 21 ± 1 °C. In each case, an increase in transmembrane pressure resulted in a corresponding linear increase in flux, due to the augmented driving force. This observation is consistent with Darcy's law (Sui et al., 2022). Moreover, the findings indicated that an increase in ethanol concentration within the hydroalcoholic solution corresponded to a decrease in permeate flux across all pressure levels for each utilized membrane. The decreased in the flux can be attributed to various factors. Ethanol might induce physical changes of the membranes due to variations in polymer chain mobility caused by the organic solvent, resulting in membrane swelling and a change in the membrane's free volume (Tarleton et al., 2006; Verhoef et al., 2008). A significant swelling might lead to an increase of the pore radius together with an increase of the membrane thickness. According to Nguyen et al. (2020), at low alcohol concentration the slight increase of both the membrane thickness and the pore are compensated resulting to an unexpected roughly stable membrane resistance. At high alcohol level, the membrane thickness increase impact overcame that of the pore radius increase so lowering the membrane performance. Additionally, changes in the solution's properties, such as its osmotic pressure, can play a pivotal role. Increased ethanol concentrations may induce a substantial osmotic pressure difference between the feed and permeate sides of the membrane, thereby opposing the fluid flow driving force across the membrane and consequently reducing the flux. As expected, white wines show the lowest membrane flux owing to their more intricate matrix, including macromolecules. These compounds of considerable

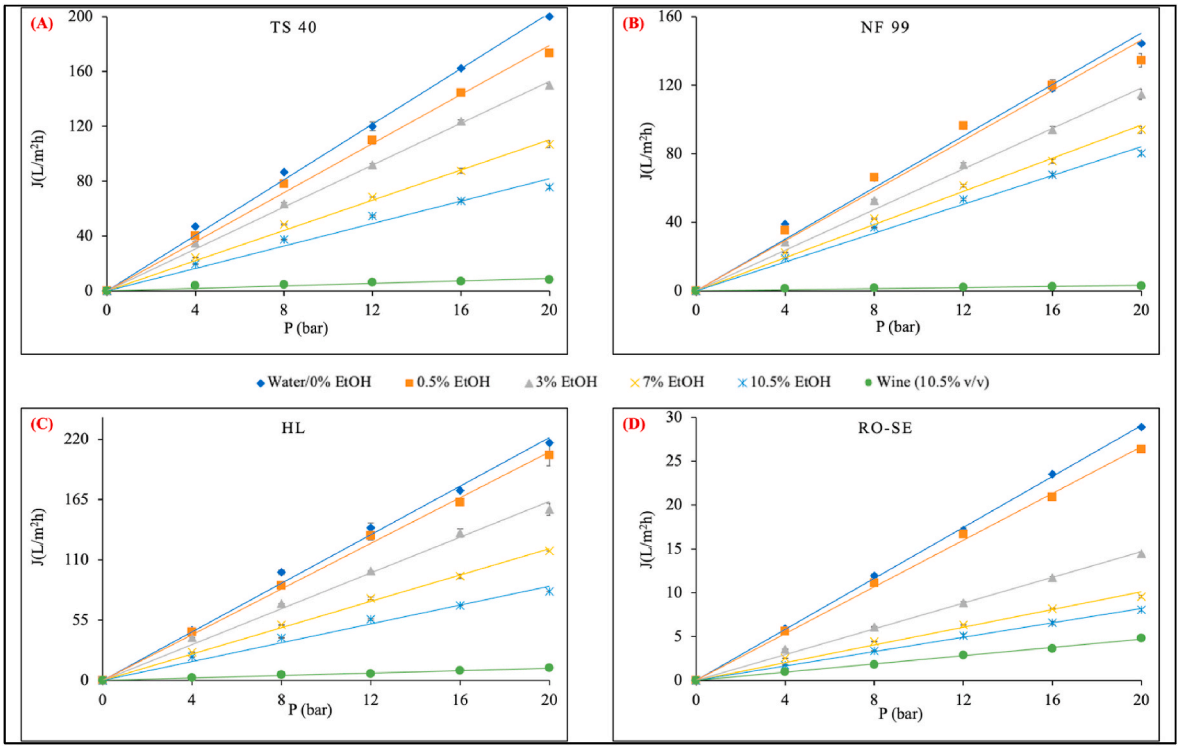

Fig. 2. Permeate flux as a function of pressure for different membranes. (A) TS40, (B) NF 99 membrane; (C) HL membrane; (D) RO-SE membrane (J, Permeate flux; P, operating pressure).

molecular size intensify the membrane fouling effect through concentration polarization and adsorption onto the membrane material, consequently resulting in reduced permeate fluxes (Giacobbo et al., 2018; Nghiem & Hawkes, 2007). Similar results were found by, Moreira et al. (2017) which compared permeation flux values of white and red wines with those of hydroalcoholic solutions with ethanol concentrations of 8, 10, 12, 14, and 16% (v/v) in a NF process performed with a self-made cellulose acetate membrane. At 15 bar, permeation fluxes decreased from 4.3 to 3.4 kg/m^{2}h, with an increase of ethanol concentration from 8 to 16% (v/v). For white and red wines average permeation fluxes resulted of the order of 2.7 and 1.7 kg/m^{2}h, respectively. On the other hand, researchers explored the effect of temperature during membrane filtration of wine: i.e. Banvolgyi et al. (2016) found that increasing the temperature from 20 to 40 °C resulted in an increase in permeate flux from 14.2 to 26.3 L/m^{2}h during the partial dealcoholization of red wine (Egri Cuvée, 2011 vintage) using a NF membrane. The increase of flux by increasing temperature is primarily due to the viscosity effect (Saleh et al., 2006).

Table 2 summarizes data of permeability obtained with selected membranes for both hydroalcoholic solutions and white wine. The results indicated that an increase in the ethanol concentration of the hydroalcoholic solution leads to a decrease in the permeability of each membrane used. The HL membrane exhibited the highest permeability (4.30–11.08 L/m^{2}hbar), while the RO-SE membrane shows the lowest, ranging between 0.41 and 1.45 L/m^{2}hbar. Furthermore, when wine was used as feed solution membrane permeability resulted much lower than the hydroalcoholic solutions for all the selected membranes with the NF99 membrane exhibiting the lowest value (0.16 L/m^{2}hbar). Similarly, Labanda et al. (2013) observed a decrease in permeability of a polypiperazine polyamide composite NF membrane with a MWCO of 65 Da (from 41.08 to 18.05 L/m^{2}hbar) when increasing the ethanol concentration of water–ethanol mixtures from 0 to 13% v/v which was attributed to the partial retention of ethanol molecules.

To assess membrane fouling and changes in water permeability after wine concentration, the pure water flux was measured at 4, 8, 12, 16, and 20 bar. The fouling index, calculated on the basis of water permeability data, resulted of 22.5%, 24.4%, 32.7%, and 43.4% for RO-SE, NF99, TS40, and HL membranes, respectively. Ivić et al. (2021a) reported fouling indexes slightly higher for polyamide NF and RO membranes (26% and 56%, respectively) in flat-sheet configuration employed for concentrating phenolic compounds of Cabernet Sauvignon red wine. Authors reported also that applied pressures influenced membrane fouling of NF with measured values from 28.59% to 31.45% in a range of pressures between 25 and 55 bar. It is well-established that interactions between membranes and feed constituents, coupled with physiochemical interactions among solutes, play a pivotal role in membrane fouling, particularly when organic materials are employed (Ulbricht et al., 2009).

Table 2
Impact of hydroalcoholic solutions and wine on membrane permeability (L/m^{2}hbar).

Hydroalcoholic solution and wine (alcohol % v/v)	Membrane type			
	NF 99	TS 40	HL	RO-SE
0	7.52	10.14	11.08	1.45
0.5	7.31	8.95	10.42	1.33
3	5.93	7.65	8.17	0.73
7	4.83	5.51	6.02	0.50
10.5	4.21	4.09	4.30	0.41
Wine (10.5)	0.16	0.46	0.56	0.23

3.2. Permeate flux and ethanol concentration in permeate and retentate samples

White wine and hydroalcoholic solution (HS) of 10.5% (v/v) were concentrated using the selected membranes under controlled conditions of 20 bar and 21 ± 1 °C, up to a volume reduction factor (VRF) of 4. Fig. 3 shown the variations in both permeate flux and TSS in wine and of HS on the permeate side throughout membrane filtration process. The initial permeate flux gradually decreased over processing time (Supp. Fig. 1) and increasing in VRF (Fig. 3). Specifically, among all the membranes used for wine as a feed, TS 40 showed the highest (45.6 L/m^{2}h) initial permeate flux, while RO-SE exhibited the lowest (5.79 L/m^{2}h) flux. In case of HS, the HL membrane displayed the highest (92.83 L/m^{2}h) initial permeate flux, whereas the RO-SE membrane exhibited the lowest (8.25 L/m^{2}h) flux. The decrease in the permeate flux can be attributed to concentration polarization phenomena which leads to an increase in osmotic pressure near the membrane-solution interface so decreasing the available driving force as well as to the retention of compounds on the membrane surface. In the case of wine, permeate flux reductions at VRF 4 with respect the initial value was approximately 21%, 42%, 48%, and 79% for NF 99, TS 40, HL, and RO-SE, respectively. Conversely, when using the hydroalcoholic solution as feed, the permeate flux reductions at VRF 4 were approximately 18, 21, 22, and 18% for NF 99, TS 40, HL, and RO-SE, respectively. The RO process required a longer duration to concentrate the wine at a VRF of 4 compared to the NF membranes, which achieved concentration in approximately 3.4 h (Refer Supp. Fig. 1). This could be attributed to the smaller pore sizes and lower molecular weight cut-off (MWCO) (<100 Da) of the RO membrane with respect the NF membranes. For both NF and RO membranes the TSS on the permeate side increased with the process time. At the end of the process at VRF 4, the TSS reached approximately 5.0 °Brix in the case of the NF 99 membrane and 5.2 °Brix for the other membranes used. For NF membranes permeate fluxes resulted higher than those obtained by Banvolgyi et al. (2016) in the partial dealcoholization of red wine (Egri Cuvée with an initial alcohol content of 12.0%, v/v) with a polypiperazine-amide membrane having a MWCO of about 500 Da (Trisep XN45). For this membrane authors reported average fluxes of 9 L/m^{2}h at 20 bar and 20 °C. Permeate fluxes of NF membranes were also higher than those reported by Ivić et al. (2021a) which evaluated the use of NF and RO membranes in the concentration of Cabernet Sauvignon red wine variety under different operating conditions. Indeed, at 25 bar and 20 °C average permeate fluxes were lower than 10 L/m^{2}h.

The performance of the selected membranes was also assessed in relation to the concentration of ethanol on both permeate and retentate side. An ideal membrane for producing low-alcohol wine can be determined by its high permeate flux, low ethanol content on the retentate side, high ethanol content on the permeate side, and the dealcoholized wine exhibiting non-significant changes in volatile and non-volatile profiles compared to the original wine (Calvo et al., 2022; Catarino & Mendes, 2011). The wine dealcoholization was performed using diafiltration as explained in section 2.3. Fig. 4A and B show the ethanol concentration on the permeate and retentate sides of both wine and 10.5% v/v hydroalcoholic solution (HS) at VRF4. The ethanol concentration measured in wine permeate samples at different diavolumes (i.e., the relative volume of a diafiltration buffer compared to the filtrate: four cycles for NF membrane and two cycles for the RO membrane) are also reported. The NF membranes showed similar levels of ethanol content on their permeate side when utilizing the hydroalcoholic solution as a feed. On the other hand, the RO permeate showed a lower ethanol content as expected in relation to the lower pore size of the RO-SE membrane as compared to other membranes. However, in agreement with data reported by Labanda et al. (2013), these results cannot be attributed exclusively to the different membrane characteristics but also to solvent–membrane interactions.

When wine was used as feed solution, the HL membrane exhibited

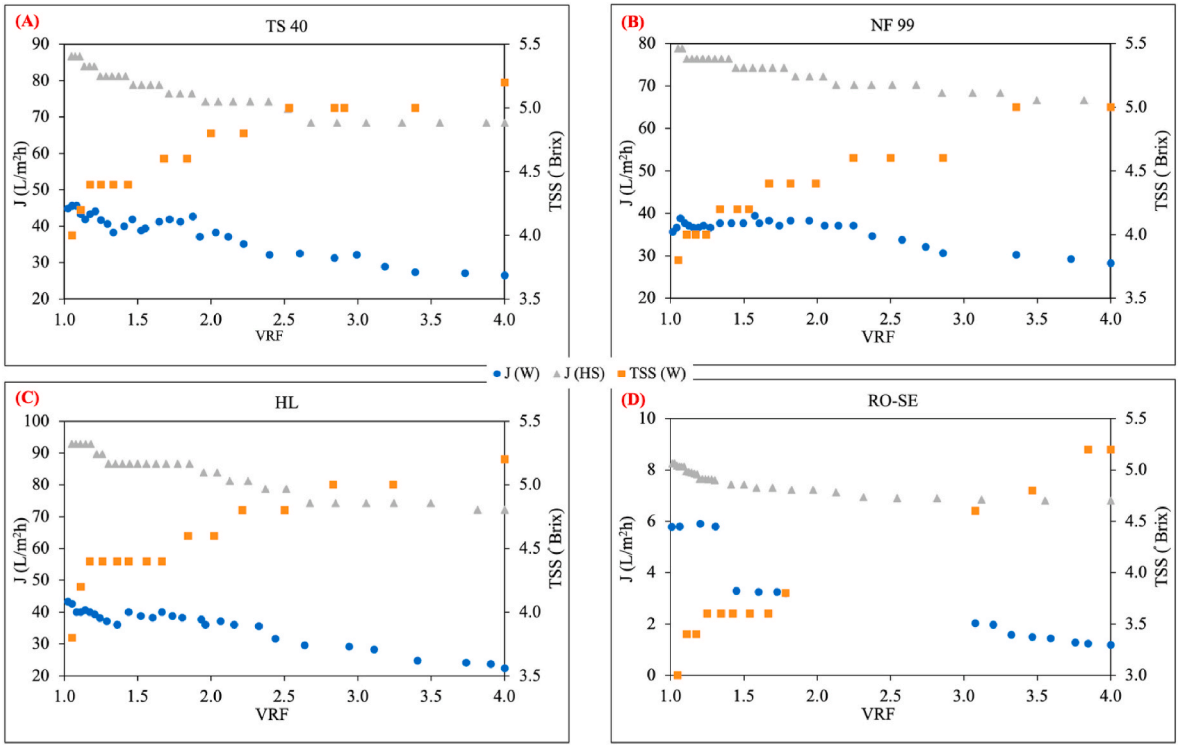

Fig. 3. Variation of permeate flux and total soluble solids on the permeate side during concentration of white wine and hydroalcoholic solution (HS) of 10.5 % v/v till volume reduction factor (VRF) 4; (A) TS 40 membrane; (B) NF 99 membrane; (C) HL membrane; (D) RO-SE membrane (J, permeate flux; W, wine; HS, hydroalcoholic solution; TSS, total soluble solids; VRF, volume reduction factor).

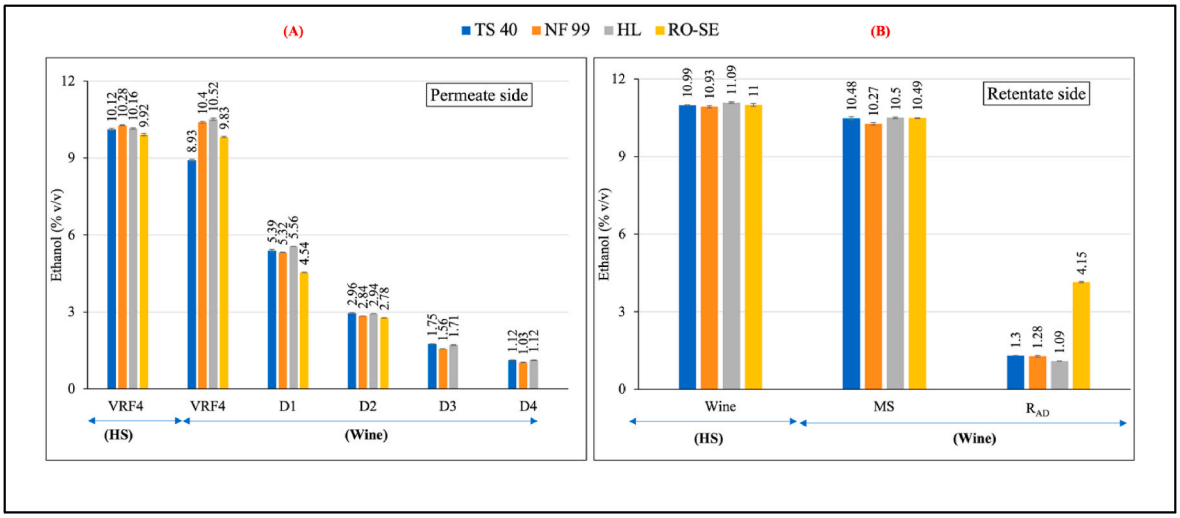

Fig. 4. Ethanol concentration in permeate (A) and retentate (B) side for hydroalcoholic solution of 10.5% v/v and wine at volume reduction factor of 4 and at different diafiltration volumes (VRF, volume reduction factor; D1, D2, D3, D4, diafiltration volume; R_{AD}, retentate after diafiltration process; HS, hydroalcoholic solution).

the highest ethanol content (10.52% v/v) on the permeate side and the lowest ethanol content (1.09% v/v) in the retentate after the diafiltration process (R_{AD}) compared to the other membranes used.

3.3. Physicochemical characterization of products

The physicochemical and color parameters of the original wine, retentate and permeate samples at VRF 4, as well as of the dealcoholized wine obtained after diafiltration process, are reported in Table 3. As expected, the concentration of most of wine components including reducing sugar, dry extract, glucose, fructose, total acidity, malic acid, and citric acid increased in the retentate samples at VRF 4 and after the diafiltration process for each investigated membrane. The concentration of dry extract increased from 27.3 to 84.3 g/L after concentrating the wine at VRF 4 using the RO-SE membrane. A higher concentration of total acidity in the retentate in comparison to NF membranes was also observed for this membrane. Volatile acidity decreased in the retentate samples for all selected membranes except for the HL membrane when compared to the initial content in the wine (0.31 g/L). The highest concentration of volatile acidity was measured in the retentate of the HL

Table 3
Physicochemical parameters of the original wine, retentate and permeate after 4-times volume reduction factor (VRF 4) and dealcoholized wine obtained after diafiltration process.

Parameter	W	RO-SE			HL			TS 40			NF99		
		At VRF4		AD-2	At VRF4		AD-4	At VRF4		AD-4	At VRF4		AD-4
		R	P	R_{AD}	R	P	R_{AD}	R	P	R_{AD}	R	P	R_{AD}
Alcohol (% v/v)	10.93	11.00	9.83	4.15	11.09	10.52	1.09	10.93	8.93	1.30	10.99	10.39	1.28
Reducing sugar (g/L)	7.82 ± 0.05^{g}	23.46 ± 0.17^{c}	3.16 ± 0.03^{hi}	9.18 ± 0.29^{f}	24.86 ± 0.21^{b}	2.36 ± 0.33^{ij}	28.77 ± 0.07^{a}	17.18 ± 0.35^{d}	3.42 ± 0.06^{h}	17.52 ± 0.38^{d}	23.23 ± 0.45^{c}	1.54 ± 0.19^{j}	13.67 ± 0.25^{e}
Dry extract (g/L)	27.35 ± 0.05^{i}	84.27 ± 0.01^{a}	9.18 ± 0.05^{l}	27.93 ± 0.18^{h}	72.94 ± 0.01^{c}	10.36 ± 0.08^{k}	61.62 ± 0.02^{d}	56.46 ± 0.14^{e}	14.82 ± 0.08^{j}	38.83 ± 0.18^{f}	74.68 ± 0.07^{b}	9.62 ± 0.02^{l}	31.06 ± 0.38^{g}
Glucose (g/L)	4.59 ± 0.20^{g}	10.07 ± 0.23^{cd}	3.17 ± 0.07^{h}	7.25 ± 0.20^{f}	11.31 ±0.11^{b}	2.22 ± 0.17^{i}	17.44 ± 0.30^{a}	8.34 ± 0.06^{e}	3.26 ± 0.15^{h}	11.62 ± 0.07^{b}	10.46 ± 0.01^{c}	2.06 ± 0.06^{i}	9.75 ± 0.29^{d}
Fructose (g/L)	3.7 ± 0.23^{g}	12 ± 0.13^{b}	1.91 ± 0.09^{h}	4.91 ± 0.15^{f}	12.5 ± 0.03^{b}	1.46 ± 0.18^{hi}	14.01 ± 0.25^{a}	8.77 ± 0.18^{d}	1.68 ± 0.03^{h}	8.96 ± 0.10^{d}	11.9 ± 0.13^{c}	0.99 ± 0.02^{i}	7.44 ± 0.18^{e}
Total acidity (g/L)	5.6 ± 0.01^{g}	15.28 ± 0.01^{a}	2.35 ±0^{k}	5.41 ±0^{h}	9.82 ± 0.01^{c}	3.71 ± 0.01^{j}	5.9 ± 0.01^{f}	9.03 ± 0.01^{d}	4.13 ± 0.01^{i}	6.14 ± 0.03^{e}	10.69 ± 0.02^{b}	3.78 ± 0.04^{j}	5.56 ± 0.01^{g}
Volatile acidity (g/L)	0.31 ± 0.01^{bcd}	0.26 ±0^{ef}	0.33 ±0^{ab}	0.3 ± 0.01^{cd}	0.34 ± 0.01^{a}	0.28 ± 0.01^{de}	0.19 ± 0.01^{f}	0.29 ± 0.01^{cd}	0.32^{a}±0^{bc}	0.06 ± 0.01^{g}	0.28 ± 0.01^{de}	0.33 ± 0.01^{ab}	0.01 ±0^{h}
Malic acid (g/L)	1.15 ± 0.03^{f}	3.71 ± 0.04^{a}	0.35 ± 0.04^{h}	1.05 ± 0.06^{f}	2.83 ± 0.04^{b}	0.42 ± 0.03^{h}	1.82 ± 0.04^{d}	2.01 ± 0.01^{c}	0.87 ± 0.01^{g}	1.46 ± 0.01^{e}	2.81 ± 0.07^{b}	0.49 ± 0.01^{h}	1.91 ± 0.03^{cd}
Lactic acid (g/L)	1.02 ± 0.01^{c}	2.11 ± 0.05^{a}	0.56 ± 0.01^{f}	1.08 ± 0.06^{c}	1.53 ± 0.04^{b}	0.74 ± 0.03^{de}	1 ± 0.08^{c}	1.1 ± 0.04^{c}	0.94 ± 0.02^{cd}	0.65 ±0^{e}	1.56 ± 0.04^{b}	0.64 ± 0.01^{e}	0.76 ± 0.11^{de}
Citric acid (g/L)	0.38 ± 0.01^{fg}	1.72 ± 0.01^{a}	0.13 ± 0.04^{h}	0.41 ± 0.01^{ef}	1.21 ± 0.01^{b}	0.13 ± 0.01^{h}	0.57 ± 0.02^{d}	1.04 ± 0.01^{c}	0.32 ± 0.02^{g}	0.58 ±0^{d}	1.26 ± 0.01^{b}	0.15 ± 0.01^{h}	0.45 ± 0.02^{e}
Tartaric acid (g/L)	1.48 ± 0.01^{f}	1.44 ± 0.08^{f}	0.2 ± 0.03^{h}	1.75 ± 0.01^{e}	1.39 ± 0.04^{f}	0.14 ± 0.09^{h}	4.18 ± 0.05^{ab}	3.24 ± 0.04^{d}	0.76 ± 0.01^{g}	4.09 ± 0.03^{b}	3.52 ± 0.02^{c}	0.29 ± 0.02^{h}	4.3 ± 0.09^{a}
Succinic acid (g/L)	0.51 ± 0.03^{bcd}	1.11 ± 0.03^{a}	0.29 ± 0.07^{efg}	0.72 ± 0.02^{b}	0.46 ± 0.04^{cde}	0.53 ± 0.01^{bcd}	0.15 ± 0.13^{fgh}	0.35 ± 0.04^{def}	0.49 ± 0.02^{cde}	0.12 ± 0.05^{gh}	0.34 ± 0.05^{def}	0.58 ± 0.06^{bc}	0.01 ± 0.01^{h}
pH	3.42 ± 0.01^{d}	3.02 ± 0.01^{g}	3.42 ±0^{d}	3.12 ± 0.01^{f}	3.62 ± 0.01^{b}	3.22 ± 0.01^{e}	3.73 ± 0.01^{a}	3.51 ± 0.01^{c}	3.21 ± 0.02^{e}	3.64 ± 0.02^{b}	3.73 ± 0.03^{a}	3.19 ±0^{e}	3.71 ± 0.03^{a}
A420nm	0.18±0^{e}	0.42 ± 0.01^{a}	0.04 ±0^{h}	0.16±0^{e}	0.42 ±0^{ab}	0.10±0^{g}	0.41 ±0^{ab}	0.32 ±0^{d}	0.05±0^{h}	0.39 ±0^{bc}	0.38±0^{c}	0.13±0^{f}	0.43 ± 0.02^{a}
A520nm	0.06±0^{f}	0.06±0^{f}	0.01 ± 0.01^{h}	0.03 ±0^{g}	0.12±0^{c}	0.04±0^{g}	0.08 ±0de	0.09 ±0^{d}	0.02±0^{gh}	0.15 ±0^{b}	0.09±0^{d}	0.08 ±0de	0.19 ± 0.01^{a}
A620nm	0.03 ±0de	0.01±0^{f}	0.01 ± 0.01^{f}	0.00±0^{f}	0.06±0^{c}	0.03 ±0de	0.01 ±0^{ef}	0.04 ±0^{d}	0.02±0^{def}	0.10 ±0^{b}	0.03±0de	0.07 ±0^{c}	0.14 ± 0.01^{a}
Color Intensity (420 + 520+620 nm)	0.27 ±0^{d}	0.45 ± 0.02^{c}	0.06 ± 0.02^{f}	0.20±0^{e}	0.60 ±0^{b}	0.16 ± 0.01^{e}	0.51 ± 0.01^{c}	0.45 ± 0.01^{c}	0.09±0^{f}	0.65 ±0^{b}	0.49±0^{c}	0.28 ± 0.01^{d}	0.76 ± 0.05^{a}
Color Tone (420nm/520 nm)	2.90 ± 0.07^{abc}	7.53 ± 0.44^{a}	6.92 ± 4.36^{ab}	5.49 ± 0.13^{abc}	3.51 ± 0.03^{abc}	2.75 ± 0.14^{abc}	5.19 ± 0.31^{abc}	3.49 ± 0.02^{abc}	2.10 ± 0.20^{bc}	2.61 ± 0.04^{bc}	4.44 ± 0.08^{abc}	1.60 ± 0.02^{c}	2.33 ± 0.08^{bc}
Chroma	13.09 ± 0.04^{g}	30.56 ± 0.14^{a}	5.24 ± 0.19^{j}	15.16 ± 0.06^{f}	26.28 ± 0.04^{c}	9.24 ± 0.08^{h}	29.95 ± 0.04^{b}	21.0 ± 0.16^{e}	4.15±0^{k}	21.57 ± 0.05^{d}	26.29 ± 0.11^{c}	7.26 ± 0.01^{i}	21.07 ± 0.23^{e}
Hue (°)	97.4±0^{e}	92.82 ±0^{g}	107.1 ±0^{b}	101.4 ±0^{d}	97.4±0^{e}	107.3 ± 0.41^{b}	96.55 ± 0.40^{f}	96.26 ±0^{f}	109.4±0^{a}	97.98 ±0^{e}	97.4±0^{e}	105.4 ±0^{c}	97.98 ±0^{e}
L^*	94.8 ±0^{cd}	97.25 ± 0.49^{b}	98.9 ± 0.42^{a}	97.15 ± 0.07^{b}	90.4±0^{f}	96.1 ± 0.35^{bc}	93.8 ± 0.28^{de}	93.1 ± 0.14^{e}	97.5 ± 0.07^{ab}	87.8 ±0^{g}	93.5 ± 0.07^{de}	92.65 ± 0.07^{e}	84.85 ± 1.20^{h}
a^*	−1.73 ±0^{bc}	−1.58 ± 0.05^{ab}	−1.53 ± 0.07^{ab}	−2.99 ± 0.01^{f}	−3.35 ± 0.10^{g}	−2.76 ± 0.01^{e}	−3.44 ± 0.08^{g}	−2.23 ± 0.04^{d}	−1.39 ± 0.01^{a}	−2.94 ± 0.04^{ef}	−3.38 ± 0.08^{g}	−1.93 ± 0.04^{c}	−2.96 ± 0.01^{ef}
b^*	12.97 ± 0.04^{g}	30.53 ± 0.15^{a}	5.01 ± 0.18^{j}	14.86 ± 0.07^{f}	26.06 ± 0.02^{c}	8.82 ± 0.09^{h}	29.75 ± 0.03^{b}	20.88 ± 0.16^{e}	3.92 ± 0.01^{k}	21.37 ± 0.05^{d}	26.07.10^{c}	7.00 ± 0.02^{i}	20.87 ± 0.22^{e}

VRF, volume reduction factor; W, original wine; R, retentate; P, permeate. R_{AD} denotes the retentate after diafiltration (AD-2 after two discontinuous diafiltration and AD-4 after four discontinuous diafiltration). Different letters indicate significant differences among samples in each case ($p < 0.05$, Tukey test).

membrane at VRF 4 (0.34 g/L). Similar results were reported by, Ivić et al. (2021b) in the concentration of Cabernet Sauvignon red wine with NF and RO polyamide membranes (RO98pHt M20 and NF M20 from Alfa Laval) in flat-sheet configuration. Authors found higher reducing sugars content and total acidity in both NF and RO retentates than in initial wine as well a decrease of volatile acidity during both processes as a result of membrane permeability to acetic acid that is the predominant compound of volatile acids. Similarly, Moreira et al. (2017) found higher concentrations of total acidity, total dry matter, and lactic acid in white wines, such as Óbidos and Palmela, after concentration with a laboratory-made NF membrane having a 90% NaCl rejection, attributable to the concentration effect.

After diafiltration, the pH of the resulting retentate showed a slight increase compared to the original wine pH (3.42) for all NF membranes. Conversely, a slight reduction of the pH was observed in the retentate of the RO membrane. The concentration of lactic acid in the retentate was higher compared to the original wine after VRF 4. Following diafiltration, there was no significant change in the case of RO-SE and HL membranes, but a decrease was observed with TS40 and NF 99 membranes.

The color parameters, including A_{420nm}, A_{520nm}, A_{620nm}, intensity, tone, chroma, hue, L^*, a^*, and b^*, were measured for the original wine and the retentates obtained from each membrane process (refer to Table 3). The results revealed a significant increase in the absorbance of the retentate at 420 nm and 520 nm, as well as in intensity, throughout each membrane process compared to the original wine. The retentate

obtained after diafiltration using NF 99 membrane shows the highest values for A_{420nm} (0.43), A_{520nm} (0.19), and intensity (0.76) among all samples. This increase contributed to a richer and darker appearance of the wine, attributed to the higher concentration of color compounds (Pérez-Magariño & González-Sanjosé, 2003).

The chroma and tone value increased remarkably in the retentate samples of all investigated membranes; in particular, the utmost values for chroma (30.56) and tone (7.53) were observed for the retentate at VRF 4 obtained with the RO-SE membrane. The increase in the chroma value indicates that the membrane concentration process resulted a more saturated color compared to the initial wine. The hue angle (°) showed a slight decrease in the retentate obtained from each membrane, except for the retentate obtained through the RO-SE membrane after the diafiltration process. Moreover, the L^* value decreased in the treatment with NF membranes, resulting in reduced wine brightness. Unexpectedly, the retentate obtained with the RO-SE membrane exhibited an increase in the L^* value compared to the original wine. In addition, the b^* value increased, while the a^* value decreased during the process. The wine concentrated by RO-SE at VRF 4 exhibited the highest b^* value (30.53) among all the samples. The rejection of selected membranes towards various components of white wine is depicted in Fig. 5. The ethanol rejection for HL and NF99 membranes were of 5.14% and 5.46%, respectively. Higher ethanol rejections were measured for RO-SE and TS 40 membranes (10.64 and 18.30 respectively). HL and NF 99 membranes exhibited significantly higher retention percentages of reducing sugars, glucose, fructose, and tartaric acid, but demonstrate lower retention for alcohol compared to other membranes. On the other hand, the RO-SE membrane showed the highest retention for compounds like, citric acid, lactic acid, malic acid, total acidity, and dry extract. These findings were in agreement with those of Catarino and Mendes (2011) which evaluated the performance of different NF and RO for producing wine with low alcohol content. Among the investigated membranes NF membranes with MWCO of 200 Da (including the NF99 used in this study) showed superior efficacy in alcohol removal from wine. This was attributed to their excellent permeability to ethanol and effective rejection of aroma compounds, leading to the production of dealcoholized wines with favorable sensory characteristics.

The retention of individual compounds depends on their molecular weight and polarity, membrane molecular weight cut-off (MWCO), membrane fouling index, and resistance (Koo et al., 2013). The higher rejection measured for the RO membranes towards most of compounds can be attributed to its smaller pore size in comparison to NF membranes (Echavarría et al., 2012).

4. Limitations and future work

The findings of this study offer valuable insights into membrane-based wine dealcoholization processes, yet several limitations warrant consideration. Firstly, the research focused solely on a specific set of membranes and operating conditions, potentially limiting the generalizability of the results to other membrane types and larger feed volume. Additionally, the use of a specific white wine composition may not fully capture the complexities of various wines available commercially, restricting the applicability of the findings. Moreover, conducting experiments at a laboratory scale (small feed volume) may overlook potential scale-up effects and variations encountered in industrial-level production of dealcoholized wine using membrane technologies. Addressing these limitations through further research encompassing a broader range of membranes, operating conditions, and wine compositions would enhance the reliability and applicability of membrane-based wine dealcoholization processes. Additionally, the integration of modeling approaches in subsequent studies is crucial for elucidating the kinetic phenomena associated with ethanol and volatile compound removal at different stages of ethanol reduction. Such insights would further enhance understanding and optimization of membrane-based wine dealcoholization methodologies.

5. Conclusions

The concentration of ethanol within hydroalcoholic solutions is found to inversely correlate with permeate flux. In addition, increasing in the operating pressure significantly enhances permeate flux yet also exacerbate fouling. Membrane HL exhibited the highest permeate flux at various pressures, demonstrated superior permeability, followed by membranes TS40, NF 99, and RO-SE. Notably, real white wine and hydroalcoholic solutions display parallel trends in flux changes with increasing pressure, while white wine, due to its intricate matrix and higher macromolecule concentration, exhibits the lowest membrane flux.

TS 40 and RO-SE membranes at VRF 4 showed high ethanol rejections, rendering them unsuitable for dealcoholization. On the other hand, HL and NF 99 membranes demonstrate the lowest ethanol rejection, proving effective in reducing wine alcohol content. Furthermore, after four diafiltration cycles, NF membranes allowed a reduction in the alcohol content of the original wine to less than 1.3% v/v. The concentrated wine produced with HL and NF 99 membranes retains the highest percentages of reducing sugars, glucose, fructose, citric acid, and

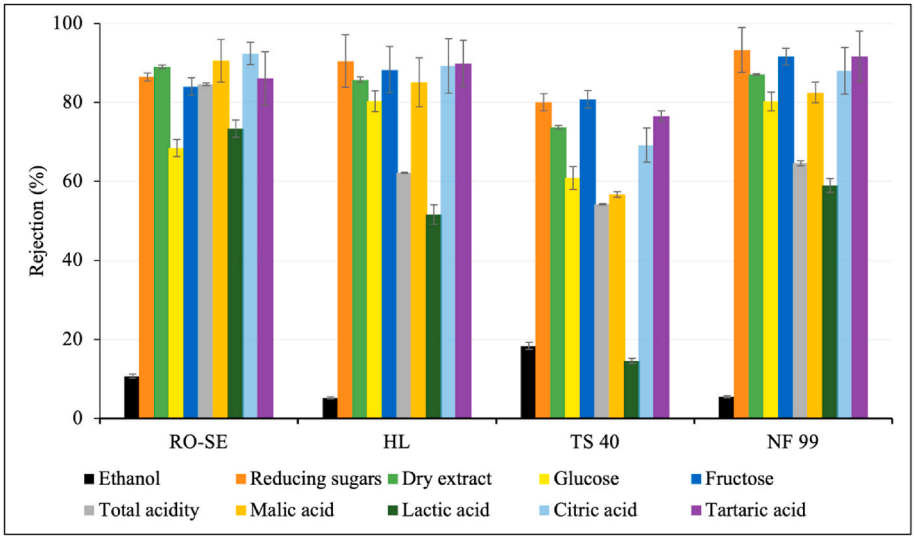

Fig. 5. Rejection of selected membranes towards specific components of white wine.

tartaric acid, but exhibits lower rejection of volatile acidity and total acidity. The concentrated wine produced from each membrane was darker in color compared to the original wine due to concentration effects. Notably, in terms of operational efficiency for ethanol removal, membranes TS40, HL, and NF 99 displayed comparable durations. Considering the overall findings, it was concluded that nanofiltration membranes HL and NF99 were the most effective among all studied membranes in producing low-alcohol wine while retaining selected wine compounds.

Further study is being conducted on an industrial-scale plant based on the assessed characteristics of the membranes mentioned earlier. The study focuses on volatile profiles, rejection of aroma compounds, and sensory analysis to gain a deeper understanding of the product's characteristics.

Metadata availability

The metadata will be made available and searchable on the AMS Acta repository (https://amsacta.unibo.it/) by recording the DOI of the article soon after publishing.

CRediT authorship contribution statement

Yogesh Kumar: Writing – original draft, Software, Methodology, Formal analysis, Data curation, Conceptualization. **Alfredo Cassano:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Data curation, Conceptualization. **Carmela Conidi:** Writing – review & editing, Methodology, Investigation, Data curation. **Arianna Ricci:** Writing – review & editing, Data curation. **Giuseppina Paola Parpinello:** Writing – review & editing, Supervision, Conceptualization. **Andrea Versari:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

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 article.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2024.116228.

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