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Evaluation of the efficiency of dialysis membranes in the wine dealcoholization process

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Abstract. This study investigated the effectiveness of dialysis membranes for the complete dealcoholization of wine (<0.50% v/v) using five different approaches. The results indicated that the sample (Dia) was ineffective due to significant water transfer into the wine, leading to considerable losses in metals, aromas, and color through the membrane. On the other hand, dealcoholized wines produced using NF+ dialysis membranes (Dia 1 and Dia 2) exhibited good retention of magnesium (Mg^{++}) and calcium (Ca^{++}), though with substantial potassium (K^{+}) losses. Furthermore, Dia 2 displayed higher chroma values (C^*_{ab}) compared to the original wine (OW). In contrast, wines dealcoholized with RO + dialysis membrane (Dia 3 and Dia 4) exhibited decreased b* and C^*_{ab} values, indicating reduced yellow coloration, while the a* value remained unchanged. Dia 4 showed no significant alteration in L* values, whereas Dia 3 exhibited lower L* values compared to OW. Principal component analysis (PCA) of the quantitative data revealed that the first two principal components (PC1 and PC2) accounted for 99.22% of the total variance. The dealcoholized samples Dia, Dia 1, and Dia 2 clustered together in the negative quadrants of both PC1 and PC2, showing no distinct association with volatile compounds. In contrast, Dia 3 and Dia 4 were positioned in the positive region of PC2, correlating with volatile compounds such as 3-methylbutanol, isobutanol, succinic acid diethyl ester, decanoic acid, and 1-hexanol.

1. Introduction

The global wine production is continuously evolving to meet the new demands and preferences of consumers. In this evolving scenario, it is important to determine which trends will be short-lived and which will remain over time. The promotion of healthier habits has encouraged consumers to try to find alternatives with low or no alcohol content [1]. The challenge for the industry is to produce an alcohol-free wine that retains the familiar aromas and mouthfeel of traditional wine but without alcohol. Ethanol is the most abundant compound in wine [2], excluding water. Numerous studies have been conducted in recent years on different technologies in the field of reducing alcohol content in wine [3–10]. A preliminary categorization was proposed by Saha et al. [4] which classified techniques based on their timing of application. These include pre-fermentation methods, such as vinicultural practices relating the use of new grape varieties with lower sugar content, clones with reduced sugar accumulation, rootstock selection, optimization of varietal selection for specific climatic conditions, early grape harvesting, and canopy management techniques like defoliation or shading. During fermentation, modified yeast strains with reduced alcohol yield and the use of enzymes for alternative metabolization, such as glucose oxidase, can be employed. These methods are promising, nevertheless they do not reduce ethanol levels to below 0.5% v/v. Achieving such low alcohol levels needs the use of physical methods used after fermentation. These methods can be further categorized into thermal processes, such as vacuum distillation, vacuum evaporation, and counter-flow distillation, as well as membrane processes like reverse osmosis, nanofiltration, osmotic distillation, pervaporation, and dialysis. Additionally, there is an extraction process category, which includes solvent extraction, carbon dioxide extraction, and absorption techniques [10]. One publication reported the use of dialysis membranes for the partial-dealcoholization of the wine [5], but so far, membrane dialysis has not been reported to be currently used in the complete dealcoholization of wine. However, in beverage technologies, it's more commonly utilized for juice concentration and beer dealcoholization [11]. This membrane has significant potential as it operates at low temperatures without requiring higher pressure, while also retaining CO_2 within the system. It's also cost-effective and adaptable for processing small volumes. The dialysis membrane is a semipermeable membrane composed of bundles of hollow fibers, typically made from cellulose derivatives or various synthetic materials like polysulfone and polyethersulfone [12]. The mechanism of dialysis involves diffusion, which facilitates the passage of ethanol between the wine and the stripping draw solution, and convection, allowing the passage of larger molecules, influencing process permeability. The efficiency of the membrane depends on the type of the dialysis membrane and the system´s pressure. The disadvantages of this method are its incapacity, until now, to prevent water passage into the wine flow of the membrane, due to a convection effect and a loss of small compounds, that move from the liquid with a high concentration, wine, to the liquid with a low concentration, water. These issues have made the use of dialysis challenging with wine. However, certain measures, such as changing the draw solution liquid, combining this membrane with other membrane technologies or heating systems, and optimizing operational parameters, could make this type of membrane suitable for producing dealcoholized wine.

The aim of the present research is to evaluate the feasibility of dialysis for the complete ethanol reduction in white wine, using several approaches and analyzing the resulting differences in key chemical parameters.

2. Materials and methods

For this study, 250 liters of processed dry white wine (produced in the winery of the Hochschule Geisenheim University) were used, made from Riesling white grape variety, grown in Rheingau (Germany), in the 2022 vintage. In this work, a dialysis membrane was evaluated for its ability to remove ethanol from:

- wine, using demineralized water as draw solution, producing dealcoholized wine (DW) referred to as Dia;
- wine, using an alcohol-free wine-based beverage as the draw solution, obtained by NF-diafiltration, producing DW referred to as Dia1;
- permeate product of NF, using demineralized water as the draw solution, and then blending back with the retentate of the NF process, producing DW referred to as Dia2;
- wine, using an alcohol-free wine-based beverage as the draw solution, obtained by RO-diafiltration, producing DW referred to as Dia3;
- permeate product of RO, using demineralized water as the draw solution, and then blending back with the retentate of the RO process, obtained DW referred to as Dia4.

Figure 1(a) shows the detailed processes of different approaches used for the production of dealcoholized wine.

2.1. Dealcoholization Procedures

The demineralized water was obtained by PureLab Option -R60, equipped with a RO, called LC119 (ELGA LabWater, Lane End Industrial Park Unit 12, HP143BY High Wycombe, Germany).

2.1.1. Diaylsis Set-UP

The dialysis set-up, shown in Figure 1(b), consists of a system where both the main solution and the draw solution are pushed through a dialysis membrane (9) in the same flow direction, originating from two different kegs (one for feed and the other for draw solution) at a pressure of 1 bar, managed by a manometer. The flow rates of the two liquids are controlled by flowmeters (3) (DK 800, KROHNE Messtechnik GmbH, Ludwig Krohne Str.5, 47058 Duisburg, Germany) at the inlets. Tubes are connected to the inlet (4) and outlet (5) of both the main solution and the draw solution, allowing for the measurement of osmotic pressure differences before and after passing through the membrane. This setup also includes four additional manometers (6), (MS 10160-WIKA Alexander Wiegand SE & Co. KG, Alexander Wiegand Str.30, 63911 Klingenberg, Germany) for system pressure verification. Two more flowmeters (7), DK800, are placed at the outlets to manage the outflow of the two liquids, allowing further regulation of their osmotic pressure. The processed liquids are collected in two separate buckets (8). The process operates discontinuously: the main solution is re-injected into the keg, and a new keg of demineralized water is introduced (one step), until the main solution (wine/permeate) is completely dealcoholized.

2.1.2. Reverse Osmosis-Nanofiltration Set-Up

The reverse osmosis-nanofiltration set-up, represented in Figure 1(c), includes a 100-litre tank (1) that supplies wine to a high-pressure pump (2), called 5CP5120 (Cat Pumps, 94th Ln NE, 55449 Minneapolis, USA). This pump feeds into a still module (3) containing either a RO or nanofiltration membrane. A manometer (4) with two valves (one with a handle (5), acting as possible by-pass, and one with a rotary knob (6)) is mounted on top of the module for pressure monitoring. The retentate is directed through a heat exchanger (7), called FL4003 (JULABO GmbH, Gerhard-Juchheim Str.1, 77960 Seelbach, Germany) regulated at 20°C, and then returned back to the tank. Another tube directs the permeate through a flowmeter (8) into a bucket (9) placed on a scale (10) for weight measurement.

2.1.3. Dialysis with Water

In the first approach, white wine was used as the main solution and demineralized water as the draw solution. Using 10 liters of wine and 80 liters of demineralized water, complete dealcoholization required 8 steps. Initially, the flowmeters were set to 10 liters of wine per 8 liters of water per hour, with pressure manually adjusted based on pressure data and mass balance control after each step. Both liquids were maintained at 15°C.

2.1.4. Dialysis with Reverse Osmosis Retentate

In the second approach, the reverse osmosis plant facilitated the permeation of alcohol and some water, while other wine components, such as aroma and flavor compounds, remained in the retentate. Through diafiltration with demineralized water, complete dealcoholization was achieved in 5 steps, processing 60 liters of wine to obtain 60 liters of dealcoholized wine. This dealcoholized wine was then used as the draw solution in dialysis to dealcoholize an additional 10 liters of wine, with both liquids at 16°C. The process took 8 steps for complete dealcoholization.

2.1.5. Dialysis with Reverse Osmosis Concentrate

In the third approach, reverse osmosis produced retentate and permeate. According to European Union (EU) regulation [13], the addition of external water to wine is prohibited. Therefore, the permeate went through further dialysis treatment to separate ethanol, allowing the reintroduction of the non-ethanol components into the concentrate. The permeate served as the main solution in dialysis, with demineralized water as the draw solution. Complete dealcoholization of the permeate required 8 dialysis steps. This cycle, combining with RO and dialysis, was repeated four times. In the first cycle, 3 dialysis steps were needed for complete dealcoholization, and 2 steps were required for subsequent cycles.

2.1.6. Dialysis with Nanofiltration Permeate

In the fourth approach, nanofiltration produced retentate and permeate. Similar to the third approach, the permeate went through dialysis for ethanol removal. Complete dealcoholization of the permeate required 8 dialysis steps. The nanofiltration-dialysis cycle was repeated twice. In the first cycle, 3 dialysis steps were necessary, and 2 steps were sufficient in the second cycle.

2.2. Wine stabilization

In all the produced dealcoholized wine, liquid SO_2 was added to achieve a free SO_2 concentration of 30 mg/L. In order to stabilize the dealcoholized wines, the wines were bottled using an instantaneous liquid heater, Getränkedurchlauferhitzer 03-0318 (Schankanlagen Koch GmbH, Dagstuhler STR.62, 66687, Wadern-Morscholz, Germany), at 62°C for 10-15 seconds in 750 mL brown glass bottles (without headspace) under screw cap closures. After that wine bottles were stored in a warehouse at temperatures between 12 and 15°C for further analyses.

2.3. Analysis

2.3.1. Chemical Analysis

The original and dealcoholized wines were analysed after 3 weeks of bottle storage following the dealcoholization process. The color parameters were measured using a photoLab® 7600 UV-VIS spectrophotometer (Xylem Analytics Germany), in the laboratory of the department of Enology, Hochschule Geisenheim University. Furthermore, total color difference (ΔEab*) between samples was obtained using the CIELAB coordinates and was calculated using the equation 1.

$$\boldsymbol{\Delta Eab*} = \sqrt{(\boldsymbol{L2*} - \boldsymbol{L1*})^2 + (\boldsymbol{a2*} - \boldsymbol{a1*})^2 + (\boldsymbol{b2*} - \boldsymbol{b1*})^2} \quad \textbf{(1)}$$

The ΔEab* represents a measure of the difference between two colors. ΔEab* is used to determine whether the human eye can visually detect the difference between two samples, using L*, a* and b* [14].

Moreover, the analysis on metals were performed by SOP-092-1 method, using an instrument called ContrAA 300 (Analytik Jena AG, Konrad-Zuse-Straße 1, 007745 Jena, Germany).

Furthermore, analysis on aroma was done at the Department of Microbiology and Biochemistry, using the method described by Schmitt et al. [15], with minor modification. It was performed with a gas chromatograph GC 5890 Series II, Hewlett Packard and a Mass spectrometer, 5972 Mass Selective Detector, Hewlett Packard.

2.4. Statistical Analysis

All measurements were conducted in triplicate, with mean values along with standard deviations. Tukey test ($p < 0.05$) and Friedman test were carried out to compare means in the ANOVA using Minitab Statistical software v.18 (State College, PA, USA). The graphs were developed with the SigmaPlot program, version 14.5.

3. Result and discussion

The research results show a clear impact of the dealcoholization process using dialysis on wine, as well as other dealcoholization methods. The aim is to evaluate which method has the least impact compared to the original wine.

3.1. Change in metal content

Figure 2(a) shows the presence of metals in the OW and in the other dealcoholized wines. The general trend observed across all analysed metals was a negative one, indicating a loss of metals in each case of dealcoholization. Magnesium (Mg^{++}), potassium (K^{+}), and calcium (Ca^{++}) were selected for analysis due to their significant impact on wine composition [16–18]. In demineralized water, these metals are present at concentrations below 1 mg/L. These elements accumulate during grape ripening, with grapes being the primary source of these meals. Various winemaking processes can influence their levels; for instance, deacidification using carbonate salts of K^+ and Ca^{++} can lead to a reduction of these metals due to the precipitation of their tartrate salts. The effects on wine properties are summarized in Table 2.

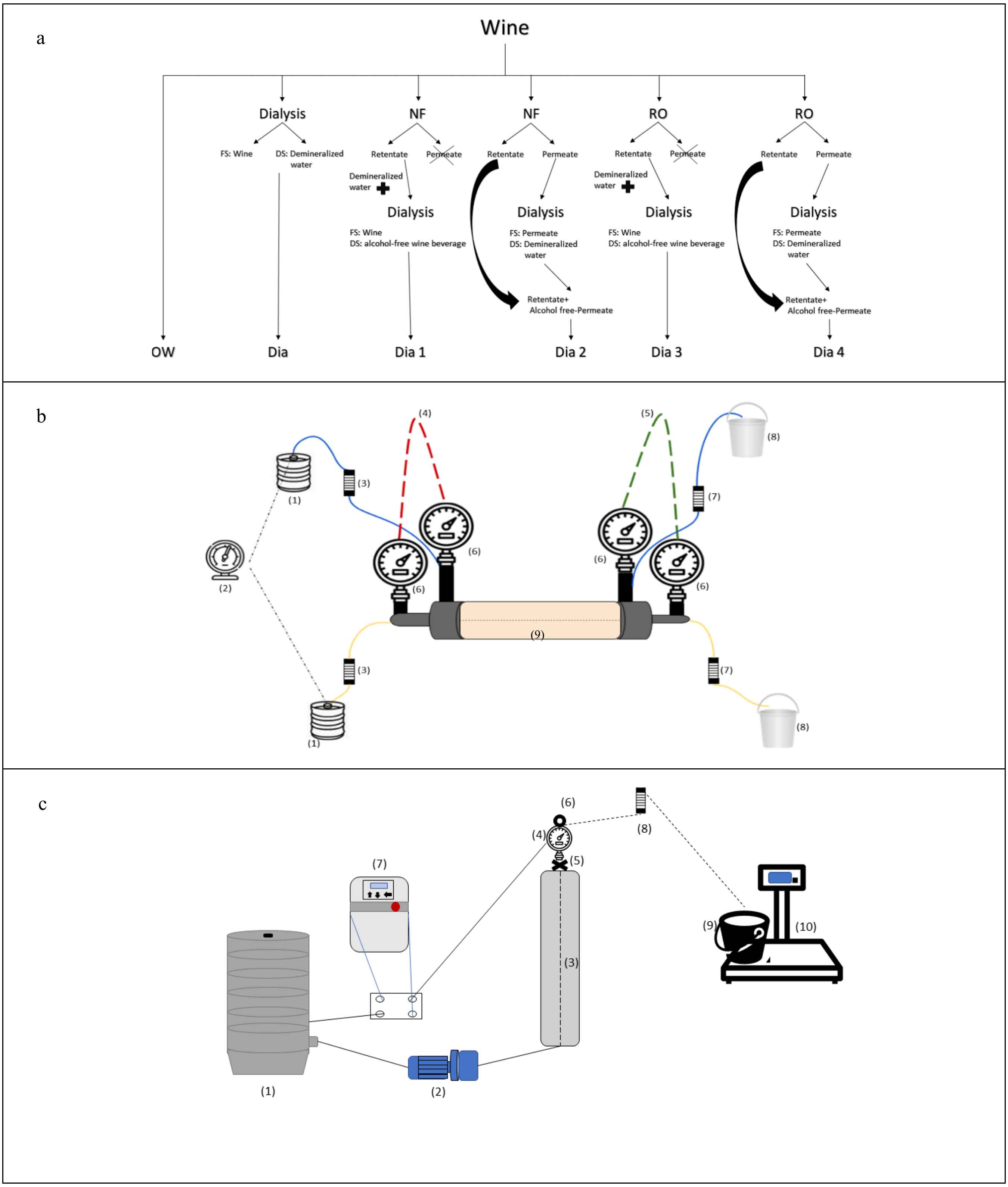

Figure 1. (a) Different approaches for the production of dealcoholized wine. Fs: Feed solution; DS: Draw solution; OW: Original Wine; NF: Nanofiltration; RO: Reverse Osmosis. (b) Dialysis set-up. (c) RO and NF set-up.

Table 1. Proprieties of the membranes used for removing alcohol from wine.

Membrane	Supplier	Type	Outer wrap	Membrane area (m2)	MWCO (kDa)	RNaCl/RMgSO4 (ppm)	P (bar)	T (°C)	pH
Dialysis	InnoSpire Technologies GmbH	Polyether Sulfone	-	4.6	10-15	-	6	40	3-11
RO (BW3022)	UNISOL Membrane Technology	Polyamide	Net wrap	7.4	80	32000 (55 bar, 25°C, pH 6.5-7)	55	50	2-10
NF (Vinopro30D)	Lenntech	Polyamide	Net wrap	7.4	150-300	2000 (8 bar, 25°C, pH 7)	80	50	3-9

- InnoSpire Technologies GmbH, Rosenweg 25, D-65510 Idstein, Germany.
- UNISOL Membrane Technology, Fritz Bothmann Str.1, 99867 Gotha, Germany.
- Lenntech B.V., Distributieweg 3, 2645 EG Delfgauw, Netherlands.

Table 2. Indicative concentration ranges, and potential impact of wine proprieties of predominant metals.

Metal	Concentration range (mg/L)	Effect on wine properties
Ca++	7-310	Precipitation with resultant pH and buffering capacity decrease
K+	125-3060	Precipitation with resultant pH change and titrable acidity decrease
Mg++	8-720	Polyphenol complexation

The data taken from [17].

All dealcoholized wines showed significant differences compared to the original wine, except for Dia 1 in terms of Mg^{++} concentration. This difference is likely to influence both the mouthfeel and stability of the wine. While most dealcoholization methods resulted in lower metal content, Dia 2 and Dia 4 retained higher levels of Mg^{++} and Ca^{++}, though not K^{+}. This might be attributed to a lesser loss of metals during the dialysis process, where the permeate was used as the primary solution and demineralized water as the draw solution, potentially resulting in a better balance between the two liquids. Potassium seems to pass through more easily, likely due to its higher initial concentration in the original wine, making it more disposed to a reduction in concentration.

Among all the dealcoholized wine samples, Dia 4 showed the closest resemblance to the original wine. The smaller pore size of the membrane likely contributed to the retention of Mg^{++} and Ca^{++} while limiting the loss of K^{+}, albeit less drastically than in other methods. On the contrary, the Dia treatment exhibited an almost complete loss of all metals. This is likely due to the differences in the composition of the main and draw solutions, which hindered equilibrium between the two liquids, promoting the migration of components from a more concentrated to a less concentrated solution. Additionally, a dilution effect may have played a role.

3.2. Color evaluation

Color is one of the key characteristics in the systematic approach to wine tasting, alongside nose and palate, and is assessed under appearance. Additionally, in recent years, color has become important to guarantee wine authenticity. For this reason, it was also used in this research to highlight differences between the wines and to draw relevant conclusions.

Figure 2(b) shows the position of the analyzed wine samples on the (a*b*) color plane within the CIELAB space. These two coordinates represent the red/green (a*) and yellow/blue (b*) components [19]. All samples, except for Dia, are located near the origin of the a* axis, with values ranging from -1.58 to -0.34 units and b* values between 4.95 and 8.48 units.

In each case, there is an increase in a* values, indicating greater browning, which is more pronounced in Dia 1 and Dia 2. For these two samples, there is also an increase in b*, which could suggest potential oxidation or aging. On the other hand, Dia 3 and Dia 4 shows a decrease in b*, indicating less yellow coloration.

Figure 2(c) shows the location of the wine samples based on their L* and C^*_{ab} values. L represents lightness, which is the visual sensation indicating how light or dark a stimulus appears. Together with a* and b*, L* defines the color of a sample [19]. C^*_{ab} is derived from a* and b* and it is a psychophysical measure indicating the degree to which a chromatic stimulus differs from an achromatic one of the same brightness [20]. The L* values are very high for all samples, ranging from 97.48 to 98.75, except for Dia and Dia 1, which both show reduced lightness. The decrease in lightness is less significant in Dia 4. Dia also exhibited notably low C*ab values.

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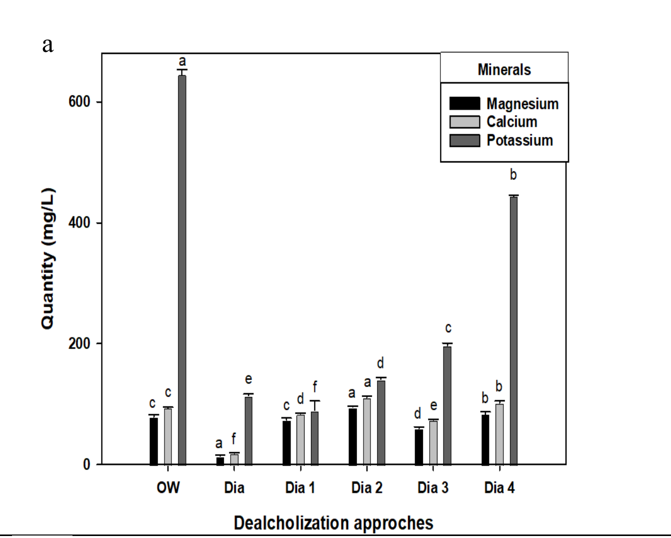
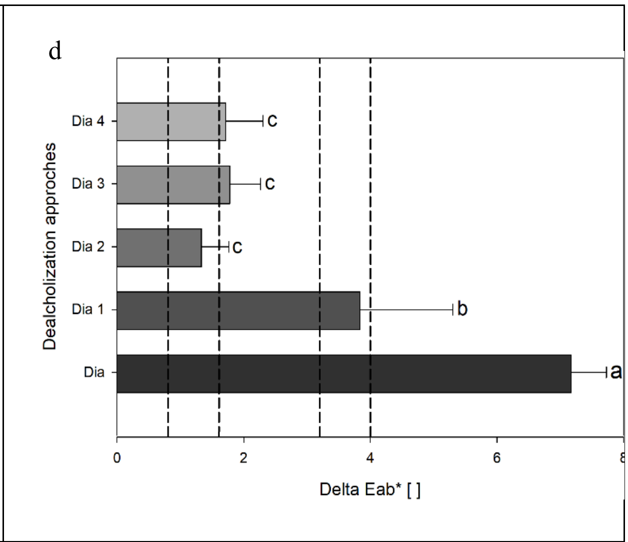
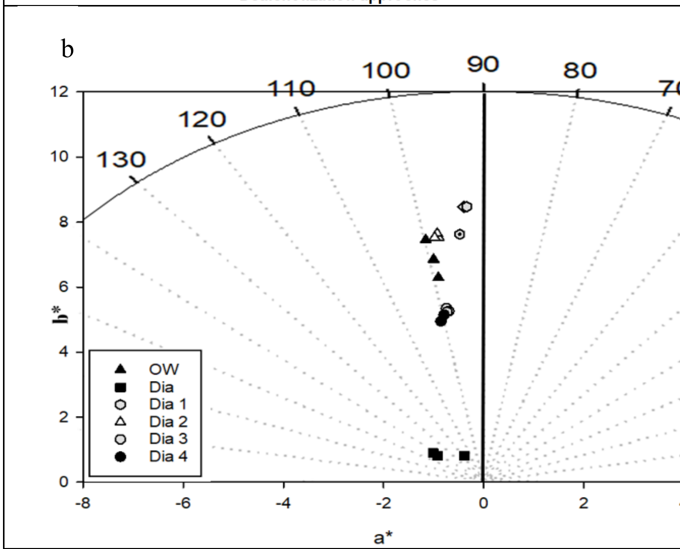
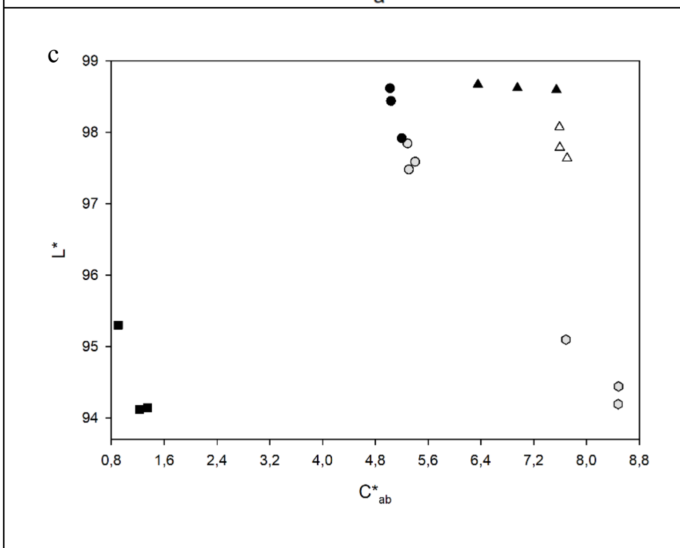






e	Interpretation aid wine
ΔE	**Evaluation**
0.0 … 0.5	No to almost no difference
0.5 … 1.0	The difference may be noticeable to a trained eye
1.0 … 2.0	Impercetible color difference
2.0 … 4.0	Perceived colour difference
4.0 … 5.0	Significant color difference, which is rarely tolerated
above 5.0	The difference is evaluated as a completely different color



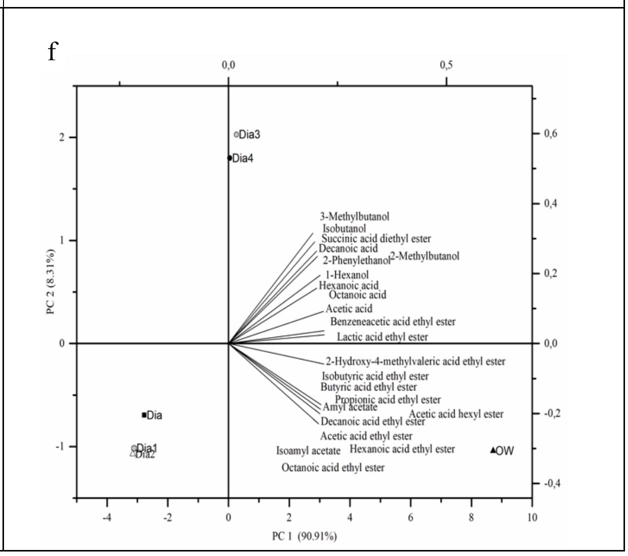


Figure 2. Dealcoholized wine composition obtain from different production approaches (a) metals; (b) representation of the wines in the color diagram (a*b*); (c) representation of the wines in the color diagram (L*C*$_{ab}$); (d) Delta E (e) Interpretation of delta Eab* score [14]; (f) PCA by plot.

Looking at Figure 2(d and e), which displays color differences, sample Dia shows a color change that is perceived as distinctly different and rarely acceptable, similarly to Dia 1. For Dia 2, the difference might be noticeable to a trained eye, while in the cases of Dia 3 and Dia 4, the color difference is imperceptible. There is no significant difference in color among Dia 2, 3, and 4.

3.3. Aroma profile variation

The loss of volatile compounds during the production of dealcoholized wines (<0.50% v/v) was investigated through Principal Component Analysis (PCA) of quantitative data (Figure 2(f)). The first two principal components (PC1 and PC2) accounted for 99.22% of the total variance. The original wine is strongly surrounded by most of the aroma compounds and positioned in the positive segment of PC1, revealing significant differences in the volatile profile of dealcoholized wines (Dia, Dia 1, Dia 2, Dia 3, Dia 4). Especially, the dealcoholized wine samples Dia, Dia 1, and Dia 2 clustered together in the negative quadrants of both PC1 and PC2, indicating a substantial alteration in their volatile composition and showing no visible association with any specific volatile compounds. On the other hand, the dealcoholized samples Dia 3 and Dia 4 are positioned in the positive region of PC2 and show correlations with volatile compounds such as 3-methylbutanol, isobutanol, succinic acid diethyl ester, decanoic acid, and 1-hexanol. The variation in results is likely due to differences in the production process. Specifically, samples Dia 3 and Dia 4 were produced using RO + dialysis membranes. The RO membrane has a smaller pore size, which retains some proportion of volatile compounds, based on its molecular size, during the dealcoholization process. In contrast, samples Dia 2 and Dia 3 were produced using NF+ dialysis membranes. The NF membrane, characterized by its larger pore size, does not retain volatile compounds to the same extent [21]. Furthermore, sample Dia exhibited no visible association with specific volatile compounds, likely due to the dilution effect caused by passing the draw solution (water) through the feed solution (wine) during the dialysis process.

4. Conclusion

The study investigated the effectiveness of different dialysis membrane approaches for complete dealcoholization (<0.50% v/v) of white wine.

In conclusion, all processes led to some degree of metal loss. However, the combination of RO with dialysis membranes (Dia 3 and Dia 4) retained Mg^{++} and Ca^{++} better than the other methods, with Dia 4 showing the closest metal profile to the original wine. The treatment involving only dialysis with demineralized water (Dia) showed significant metal loss, (Mg^{++}, Ca^{++} K^{+}) highlighting the inefficiency of this approach.

In terms of color, Dia 3 and Dia 4 samples exhibited the least alteration, maintaining characteristics similar to the original wine, after the dealcoholization process. These samples showed only minor decreases in lightness (L*) and C^*_{ab}, indicating minimal impact on the wine's appearance. In contrast, samples treated with nanofiltration (NF) and dialysis membranes (Dia 1 and Dia 2), as well as the standard dialysis method (Dia), showed more browning and noticeable color changes, which could be perceptible to consumers.

PCA results indicated that Dia 3 and Dia 4 samples clustered closer to the original wine in terms of aroma, while Dia, Dia 1, and Dia 2 showed significant deviations.

Based on these results, Dia 4 appears to be the most promising approach for dealcoholizing wine using dialysis membranes. It resulted in the least impact on color perception, retained higher levels of metals like Mg^{++} and Ca++, and showed better volatile compound retention compared to other methods. The pore size of the membrane played a key role in these outcomes, allowing for better retention of certain compounds. The findings suggest that a combined approach of dialysis and RO holds potential. Further studies should explore these methods in greater detail and investigate the possible passage of external water into the wine, which mass balance analysis could not fully detect.

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