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Evaluation of cross-flow filtration and electro dialysis in four wine varieties under industrial conditions

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Abstract

The turbidity of wines needs to be reduced through filtration, with the other advantage of performing wine stabilization. Among the available techniques, cross-flow filtration is largely applied today because it does not require conventional filter media or filter aids, it can be applied during all the stages of winemaking and shows very high efficiency. Electro dialysis consists of separating differently charged ions, by the use of selective permeable membranes under the action of an electric field. It is used to apply tartrate stabilization on wines. These two techniques were applied to two red and two white wines, respectively cv. Nero d'Avola and Syrah and Catarrato and Grillo in an experimentation performed in Sicily (Italy) to evaluate the influence of these procedures on the quality of the wines ready to be bottled. Samples of wines were analyzed to evaluate the most important quality parameters (alcohol, pH, total acidity, volatile acidity, malic acid, lactic acid, citric acid, tartaric acid, ashes, color intensity and Hue, absorbance at 420, 520 and 620 nm, polyphenols, catechins, free sulfur dioxide, total sulfur dioxide, conductivity) and the aromatic profile by gas chromatography. Red wines showed greater sensitivity to quality change after the treatments, with particular reference to color.

Key words: aromatic component; polyphenols; quality; wine stabilization.

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Introduction

Consumer demand and the highly competitive market are currently driving wineries to produce high-quality wines. The quality of a wine is determined by the physicochemical interaction of parameters such as alcohol content, sugar, density, total acidity, volatile composition, and sulfite levels, along with sensory attributes including aroma, taste, astringency, bitterness, color, turbidity, and off-odors. According to Gutiérrez-Escobar *et al.* (2021), these parameters depend on several factors, such as grape variety and pedoclimatic conditions, but above all, on winemaking techniques.

In the production of beverages such as wine, milk, and, in some cases, fruit juices, filtration plays a crucial role in the process. Before bottling, beverages must be filtered to remove sediments, impurities, and undesirable substances that can affect the flavor, appearance, and overall quality of the final product. Among the most advanced and efficient technologies used in this field is cross-flow filtration. Cross-flow filtration is a technique that uses porous membranes capable of retaining particles with sizes ranging from 0.2 to 0.4 μm . Membrane processes have become some of the most widely used techniques in the food industry over the past few decades. These processes are applied in the dairy, wine, beer, fruit juice, and sugar industries (Arend *et al.*, 2017; Bhattacharjee *et al.*, 2017; Daufin *et al.*, 2001; Tamime, 2013). In the wine sector, the use of these technologies allows for reduced production costs through automatic, continuous, and controlled processing. It also decreases energy consumption, processing time,

and wine loss (Pasechnaya *et al.*, 2023). Moreover, it contributes to wine stabilization, ensuring the absence of physical, chemical, or organoleptic alterations during a given storage period (Thoukis, 1974). The most common membrane processes include microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO) and electro dialysis (ED) (Charcosset, 2021). Cross-flow microfiltration and membrane plate filtration are the main filtration techniques used in wineries. Rosária *et al.*, (2022) investigated the impact of these two filtration processes on red wine turbidity levels, phenolic composition, chromatic characteristics and sensory profile. Ma *et al.* (2020) evaluated the influence of various clarification treatments, including the use of fining agents such as bentonite (BT) and soybean protein (SP), as well as MF and centrifugation (CF), on the volatile composition and aromatic characteristics of wine samples. All the treatments improved wine clarity. The authors reported that bentonite aging had the greatest influence on the aromatic and gustatory properties of the samples, while membrane filtration mainly affected color and aroma. SP and CF treatments achieved superior sensory quality.

One of the main causes of haze and precipitation in wine, according to Gnilomedova *et al.* (2022), is the formation of soluble potassium and calcium tartrates. To prevent tartrate deposits in wine bottles -which are generally rejected by consumers - several stabilization processes are employed. Among them, ED is a well-established and authorized technique in the agri-food industry. It involves the removal of certain ions from the wine, particularly potassium, calcium, and tartaric acid ions, contributing to the reduction of tartaric acid salt oversaturation. It is applicable to all

wine types, whether filtered or unfiltered, and stabilizes the product at room temperature while preserving its original structure and color. Tartaric stabilization is essential to achieve high-quality wines (Saint-Pierre *et al.*, 1998), ensuring clarity and quality throughout storage and commercialization. Tartaric precipitation, particularly the formation of potassium hydrogen tartrate (KHT) crystals, represents one of the most common physicochemical instabilities in wine. “Tartrate stabilization on an industrial scale of three sherry wines (“Fino”, “Medium” and “Cream”) has been carried out by means of cold treatment and ED with the objective of checking the efficacy of these techniques. This was determined by analyzing the compounds involved in the treatments and by conductivity techniques for rapid tartaric stability control (saturation temperature and Mini Contact test). It has been proven that both cold treatment and ED have imparted tartaric stabilization to all the considered wines, with the latter requiring deionization rates of 26% in Fino wine and less than 20% in Medium and Cream wines. It has been shown that the Mini Contact test is a valid method to control the efficacy of the treatments, differentiating between stable and unstable wines. The application of ED allows for the removal of cations responsible for tartaric salt precipitation (Dabare *et al.*, 2023). ED is recognized as an authorized oenological practice by the OIV (OIV-OENO 494-2012) and is approved under both European and U.S. regulations. Unlike pressure-driven processes, ED is a feasible, fast, and chemical-free technique for tartrate stabilization, as well as wine acidification and deacidification (Tsygurina *et al.*, 2022). Among all stabilization steps, tartaric stabilization before bottling -to avoid the precipitation of acid tartrate salts- is one of the most critical and widely practiced in wine-making (Martínez-Pérez *et al.*, 2020). Gómez Benítez *et al.* (2003) carried out industrial-scale tartaric stabilization of three Sherry wines (Fino, Medium, and Cream) using both cold treatment and ED. The authors demonstrated that both methods effectively ensured tartaric stabilization in all the wines tested. A deionization rate of 20% was applied for the Medium and Cream wines, and 26% for the Fino wine, to achieve adequate stability. ED employs ion-selective permeable membranes -both cationic and anionic- allowing for the removal of ionic species. Consequently, a partial removal of anions such as sulfate ions can also occur (Cabrita *et al.*, 2016). An ED unit typically consists of a central chamber containing the product to be treated (*e.g.*, wine), which is separated from the anode and cathode compartments, through which water flows, by alternating anion- and cation-exchange membranes (Martínez-Pérez *et al.*, 2020). One of the key advantages of ED is that it does not interfere with wine compounds that significantly contribute to its sensory properties (Gonçalves *et al.*, 2003). As an ion-extraction method, ED allows for the targeted removal of KHT to the extent required to achieve the desired level of tartaric stability, while also enabling the removal of calcium ions. Importantly, this technique preserves wine’s organoleptic integrity, ensuring that flavor, aroma, and other sensory characteristics remain unaffected. Based on the literature, most investigations have been conducted at laboratory or pilot scale and have primarily addressed technical feasibility, specific wine styles, or single-aspect evaluations. To date, only Gómez-Benítez *et al.* (2003) reported industrial-scale applications of ED, but their study was restricted to Sherry wines and did not provide a comprehensive assessment of quality parameters across different wine types. The present study advances this field by applying cross-flow filtration and ED stabilization to both white and red wines under real industrial conditions using innovative equipment. Specifically, we evaluate ED performance in achieving tartaric stability while also assessing its impact on key

physicochemical and sensory attributes of the wines prior to bottling. By offering one of the first systematic, multi-product assessments of ED at industrial scale, this work provides novel insights into its technological feasibility and potential as a reliable alternative to conventional stabilization practices. The experimental design was structured at industrial scale, with each treatment applied to independently processed sub-batches for every grape variety, thereby allowing evaluation of treatment performance and process-level variability under real winery operating conditions.

Materials and Methods

The trials were conducted at the “Tenute Rapitalà Spa” winery, located in the province of Palermo (Sicily, Italy). For this research work, four wines were used: two white wines from the Catarratto and Grillo varieties, and two red wines from the Syrah and Nero d’Avola varieties.

Cross-flow filtration system

Filtration was carried out using an Oenoflow XL-4 S tangential flow filter (Pall®, New York, NY, USA). This system consists of four tangential filtration modules, providing a total filtration surface area of 86 m², with a flow rate ranging from 40 to 60 hL/h. The modules are equipped with hollow fiber membranes made of PVDF (polyvinylidene fluoride) with a symmetric structure. The module housing is made of transparent polysulfone, allowing for real-time monitoring of filtrate quality and the detection of any potential issues, while the seals are made of ethylene-propylene copolymer and the casing of polypropylene. During the experiment the Oenoflow XL-4S unit was operated with PVDF hollow-fiber membranes of 0.45 µm pore size at a pressure of 1.5 bar.

All components in contact with the product are made of AISI 304 stainless steel. The collectors, piping, membranes, pumps, and instruments are mounted on a mobile frame equipped with a stainless steel mesh pre-filter to retain coarse particles, an integrated concentration and washing tank, a feed pump, a recirculation pump, a filtrate tank, a filtered wine and backwash pump, a magnetic flow meter with batch control, a Clean-In-Place (CIP) system with a 30-inch, 1-micron water filter and temperature control, automatic chemical dosing with three PLC-controlled feeding lines, a touch-screen display, safety protections for overpressure, overheating, and dry running, and a power socket for an external feed pump. The Oenoflow XL system analyzes production data and automatically adjusts operational parameters in real-time to maximize system efficiency. The system is connected to servers, allowing users to easily access their production data via the Oenoflow PRO app or web portal. Operating cycles can be configured using the patented Low Concentration Volume (LCV) function and automatic chemical dosing, enabling clarification with low operating costs without affecting wine quality.

Electrodialysis for tartaric stabilization

For the tartaric stabilization process, an ED-30 electro dialysis system (JU.CLA.S. S.r.l., Settimo di Pescantina, VR, Italy) was employed. ED is a separation technique that utilizes the driving force of an electric field acting within a system of selective membranes, which separate the ions contained in the wine and isolate the electrodes used to generate the electric field. The ED-30 unit stabilizes the wine at room temperature, without denaturing colloids or altering the phenolic composition of the wine. In addition to separating tartrate ions from potassium ions, the system also

partially removes other ionic species such as calcium and magnesium, further improving tartaric stability – especially in cases where cold stabilization is ineffective.

The ED-30 system provides the following benefits: tartaric stabilization of both white and red wines; the ability to control the amount of ions removed; the removal of other ions besides potassium and tartrate, such as calcium (ranging from 21% to 43%), magnesium (from 4% to 11%), and, to a lesser extent, iron; a different ratio of potassium and tartrate removal compared to cold stabilization, resulting in a smaller decrease in total acidity; preservation of wine's color and structure with no volume losses; processing at room temperature; no alteration of other wine parameters; zero additives; a fast and cost-effective process with no wine losses and the possibility of reusing the process water.

The system is equipped with two reactors, featuring a total power of 8 kW and a nominal voltage of 400 V. The stabilization process was performed at a flow rate of approximately 30 hL/h, with a concentrated solution (brine) waste of about 600 L/h. The ED-30 system was run at a voltage gradient of 2 V per cell with a deionization rate of 20%, in accordance with OIV guidelines and previous industrial-scale studies, ensuring effective tartaric stabilization while preserving wine quality. The process was conducted in the absence of oxygen by introducing an inert gas, such as nitrogen. The system is also equipped with two hydraulic circuits that independently supply the two types of compartments in parallel: one manifold collects all the compartments containing the treated wine, while another manifold connects all the compartments containing the concentrated solution. The system is powered by direct current via two electrodes (anode and cathode) that regulate the process and establish a potential difference of approximately 2 V per cell across each membrane. The unit includes a feed tank and pump, as well as two separate tanks for the treated wine and the concentrated solution. These tanks are equipped with level and conductivity sensors. The entire system is fully automated and controlled by a dedicated control unit that manages the wine and concentrated solution circulation sequences. The liquid circulates between the feed tank and the ED cells until the desired treatment level is reached, as determined by measuring the conductivity of the product. When the conductivity reaches the threshold established by the instability test, the stabilized wine is discharged from the circuit through a solenoid valve and automatically transferred to the collection tank. Simultaneously, a new batch of wine is introduced into the system to undergo the same stabilization process. ED enables the selective extraction of K^+ and HT^- ions required to achieve tartaric stabilization.

Sampling and analysis

For each of the four wine types, analyses were carried out after tangential flow filtration and after tartaric stabilization. Wine samples were collected at three stages: before the treatments (control), after tangential flow filtration, and finally after tartaric stabilization by ED. Table 1 summarizes the experimental treatments applied. Red wines were produced by sulfiting the grapes with 50 mg/L of SO_2 in closed stainless-steel tanks, followed by inoculation with a standard yeast strain (*Saccharomyces cerevisiae*, Fermol Cru, AEB Group, Brescia, Italy) at a dosage of 20 g/hL. Following alcoholic and malolactic fermentation, the wines were stored in stainless steel tanks under controlled conditions (temperature 18°C) and regularly monitored for free SO_2 levels. Three samples were collected for each variety and for each treatment, including the control, resulting in a total of 36 samples. From the main storage tank of each wine variety, the wine was divided into

three independent stainless-steel sub-batches prior to the application of technological treatments. Each sub-batch was processed independently under identical industrial operating conditions. Specifically, each sub-batch underwent cross-flow filtration as an independent process cycle. Subsequently, each filtered sub-batch was subjected independently to electro dialysis (ED), with the ED system treating three separate process flows corresponding to the three sub-batches. Therefore, for each variety × treatment combination, three independent process-level experimental units were obtained. Samples were independently collected from each treated sub-batch for physicochemical and chromatographic analyses.

The reported values represent the mean ±SD of three independently processed sub-batches and reflect process-related variability under industrial-scale conditions, consistently with previous studies at industrial scale (Gómez-Benitez *et al.*, 2003). Before being sent to the laboratory for physicochemical analyses, the samples were immediately bottled in 0.750 L dark glass bottles sealed with cork stoppers. Immediately after the manual filling and before applying the cork closure, nitrogen was added to remove oxygen from the bottle headspace (5 mL). All wine samples were maintained at 18°C in the dark until analysis. Analytical determinations in musts and wines were performed by Foss Integrator WineScan™. (FOSS Italy), a multi-parameter analyzer that utilizes Fourier Transform Infrared technology, scanning samples in mid-infrared wavelength range. This method enables simultaneous acquisition of multiple parameters after a single reading.

The wine determinations were alcohol (%/vol), density (g/l), sugar (g/l), pH, total acidity (g/l), volatile acidity (g/l), malic acid (g/l), citric acid (g/l), tartaric acid (g/l), K^+ (g/l), polyphenols (mg/l), ashes (g/l), RAN (readily assimilable nitrogen) (g/l) Ca^{2+} (mg/l), Cu^{2+} (mg/l), gluconic acid (g/l), methanol (g/l), CO_2 (g/l), catechins (mg/l), total sulfur dioxide TSO_2 (mg/l), free sulfur dioxide FSO_2 (mg/l). Furthermore, for red wines, the color and Hue are determined after the acquisition of visible spectra of undiluted samples using 1-mm optical path cuvettes. Color intensity (CI) was calculated as the sum of absorbance measured at 420, 520, and 620 nm ($A_{420} + A_{520} + A_{620}$ on an optical path of 10 mm) and Hue was obtained as the ratio of absorbances measured at 420 and 520 nm (A_{420}/A_{520}) following the method OIV-MA-AS2-07B.

The gas-chromatographic analyses were run on a Hewlett-Packard 5890 GC system interfaced with a HP 5973 quadrupole mass spectrometer. A HP5-MS column was used (5% diphenyl – 95% dimethylpolysiloxane 30 m × 0.2 mm. 0.25 µm film, J & W Scientific, Folsom, CA, USA). Ultra-high-purity helium (Praxair, Cleveland, OH, USA) was the carrier gas. Water and oxygen traps were installed on the carrier gas lines was employed. The column temperature was held at 40 °C for 15 min and then was increased to 220°C at 1°C/min. The carrier gas (helium) flow rate was 1 mL min⁻¹. The spectra were recorded at an ionization voltage of 70 eV and an ion source temperature of 220°C (De Pasquale *et al.*, 2006). Samples were analyzed by HS-SPME-GC-MS method with a PDMS-CAR-DVB fiber (Supelco). The fiber was manually

Table 1. Experimental treatments applied: grape varieties and treatments.

Variety	Treatment		
	Control	Cross-flow filtration	Electrodialysis
Catarratto	CC	CFC	EDC
Grillo	CG	CFG	EDG
Nero D'Avola	CNA	CFNA	EDNA
Syrah	CS	CFS	EDS

inserted in a GC inlet port equipped with a specific glass liner for SPME injection (0.75 mm i.d.). Fibers were desorbed at 250°C in splitless mode for 1 min into gas chromatograph inlet. Sample components were verified by comparison of the mass spectral data with those of authentic reference compounds. When standards were not available, the components were identified by mass spectrum matching using the NIST05 mass spectral library collection.

Statistical analysis

For each variety × treatment combination, three independent process replicates were obtained as described above. Data are reported as mean ± SD of these independent experimental units. Initially, one-way ANOVA followed by Tukey's *post-hoc* test ($p < 0.05$) was applied to compare treatments within each grape variety. To further investigate the interactions between factors, a two-way ANOVA was conducted considering variety (Catarratto, Grillo, Nero d'Avola, Syrah) and treatment (control, cross-flow filtration, electrodialysis) as independent variables, and physico-chemical or volatile parameters as dependent variables ($p < 0.05$). This approach allowed us to test not only main effects but also whether treatment responses differed across varieties. Statistical analysis was carried out using Statgraphics Centurion (Statpoint Technologies Inc., Warrenton, VA, USA).

Results and Discussion

Physico-chemical analyses

Table 2 presents the results of the physico-chemical analyses for the Catarratto variety, comparing the three treatments (CC, CFC and EDC). Not statistically significant differences were observed in the alcohol content, while pH showed a significant decrease of

approximately 0.10 units, dropping from 3.32 in the CC treatment to 3.21 in the EDC treatment. This reduction is attributed to a decrease in cations (Ca^{2+} , K^+) and anions (AT^-), with cations being reduced more easily than anions.

ED is distinguished by its ability to selectively remove the ions responsible for wine instability, acting directly on the root cause of the problem. In general, pH is lower in wines treated with ED, which can be an additional advantage for wines from warm regions, where pH is usually high and acidity is low (Martínez-Pérez *et al.*, 2020). Regarding total acidity, malic acid, and tartaric acid, significantly lower values were observed in the EDC treatment compared to CC and CFC. Specifically, total acidity values were 5.72 g/L in CC, 5.71 g/L in CFC, and 5.54 g/L in EDC. For malic acid, the values were 1.60 g/L in CC, 1.58 g/L in CFC, and 1.42 g/L in EDC. Lastly, tartaric acid levels in the EDC treatment were reduced by 0.38 g/L compared to CC. Not statistically significant differences were found among the three treatments for volatile acidity, a parameter that is essential for wine authenticity and should remain stable over time. Conversely, a significant reduction was observed in the EDC treatment compared to CC for the cationic fraction parameters K, RAN, and Ca^{2+} , with reductions of 0.31 g/L for K^+ , 9 g/L for RAN, and 8.50 mg/L for Ca^{2+} . Not statistically significant differences were observed for catechins, while in the EDC treatment, Cu^{2+} showed a significant reduction of 0.11 mg/L compared to CC. At high concentrations, this element can be harmful to human health and is present in wine due to copper-based treatments applied in vineyards. In addition, Cu^+ can catalyze oxidative reactions in wine, leading to browning in white wines and modification of aromatic compounds, thereby altering the wine's organoleptic characteristics and long-term stability (Mercanti *et al.*, 2024; Rousseva *et al.*, 2016). It has been reported that Cu^+ levels as low as 0.05 mg/l 1 Cu may strongly influence the oxidation rates of wine (Zhang and Clark, 2023). Cu^{2+} originates from multiple sources, including soil uptake, fungicide applica-

Table 2. Physico-chemical parameters measured in the wine Catarratto. The analyses were performed in triplicate for each treatment, and the mean values obtained were compared using Tukey's test at a 95% significance level (Statgraphics centurion, Statpoint Inc., USA, 2005).

Parameter	CC	CFC	EDC
Alcohol (%/vol)	12.14±0.05	12.13±0.04	12.14±0.07 ^{ns}
pH	3.32±0.02 ^a	3.31±0.04 ^a	3.21±0.02 ^b
Total dry extract (g/l)	23.35±0.41 ^a	23.40±0.47 ^a	21.03±1.19 ^b
Total acidity(g/l)	5.72±0.03 ^a	5.74±0.05 ^a	5.54±0.06 ^b
Volatile acidity (g/l)	0.21±0.02	0.20±0.02	0.21±0.02 ^{ns}
Malic acid (g/l)	1.60±0.04 ^a	1.58±0.05 ^a	1.42±0.06 ^b
Lactic acid (g/l)	0.29±0.01 ^b	0.30±0.02 ^b	0.34±0.01 ^a
Citric acid (g/l)	0.29±0.04	0.28±0.03	0.27±0.04 ^{ns}
Tartaric acid (g/l)	2.71±0.14 ^a	2.73±0.10 ^a	2.34±0.09 ^b
Potassium (g/l)	0.73±0.06 ^a	0.715±0.10 ^a	0.42±0.05 ^b
RAN (g/l)	42.50±3.54 ^a	42.50±0.71 ^a	34.00±1.41 ^b
Ca^{2+} (mg/l)	83.50±0.71 ^a	84.50±0.71 ^a	74.50±0.71 ^b
Polyphenols (mg/l)	315±14.50 ^a	316±3.60 ^a	271±17.00 ^b
Catechins (mg/l)	15.23±0.38	17.83±0.81	17.83±1.87 ^{ns}
Ashes (g/l)	2.34±0.10 ^a	2.31±0.14 ^a	1.65±0.08 ^b
Cu^{2+} (mg/l)	0.23±0.03 ^a	0.23±0.04 ^a	0.12±0.02 ^b
TSO_2 (mg/l)	112±9	112±10.58	98±17.32 ^{ns}
FSO_2 (mg/l)	40.50±0.71 ^a	39.50±0.71 ^a	31.50±0.71 ^b

Data reported are mean ± SD; ns, not significant; different letters in the row indicate statistically significant differences.

tion, contamination during production, or deliberate addition by winemakers to eliminate sulfidic off-odors (Clark *et al.*, 2015; Yue *et al.*, 2024).

A decrease of 14 mg/L in total sulfur dioxide (TSO₂) was also recorded in the EDC treatment compared to CC. Finally, a statistically significant reduction in polyphenols was observed after the tartrate stabilization process, decreasing from 315 mg/L in the CC treatment to 271 mg/L in the EDC treatment. As observed for the Catarratto variety, not statistically significant differences were found for the alcohol content in Grillo variety (Table 3). Regarding volatile acidity, total acidity, malic acid, lactic acid, and citric acid, not statistically significant variations were recorded. In contrast, pH showed a significant decrease of approximately 0.11 units, dropping from 3.29 in CG to 3.18 in EDG.

Among the cationic fractions, only K⁺ showed a significant reduction of 0.40 g/L in EDG compared to CG. Conversely, not statistically significant variations were observed for Ca²⁺, RAN, or total sulfur dioxide (TSO₂), consistent with the results obtained for catechins and Cu²⁺. The reduction of K⁺ leads to a decrease in pH and an increase in titratable acidity, improving the wine's sensory freshness and microbiological stability. Furthermore, a statistically significant reduction in polyphenols was observed, decreasing from 204 mg/L in the control (CG) to 159.70 mg/L in the electro-dialysis-treated sample (EDG).

Finally, Tables 4 and 5 report the analyses of red wines from Nero d'Avola and Syrah grape varieties, respectively. It can be observed that the alcohol content decreases, especially during ED, (Rosária *et al.*, 2022) with a variation of about 0.10% vol. in both samples. Alcohol, being a highly volatile compound, may significantly decrease during wine transfers or when using winemaking equipment such as pumps. The pH also shows a significant decrease, by 0.08 units in EDNA compared to CNA and by 0.09 units in EDS compared to CS. It is well known that pH plays an

important role in the color of wine as it affects the equilibrium between the different forms of anthocyanins (Brouillard and Delaporte, 1977). Payan *et al.* (2023) state that total acidity and pH are often associated with the freshness of wine; in particular, a decrease in Hue and an increase in color intensity (IC) are probably due to a decrease in wine pH. Volatile acidity in both wines does not show statistically significant variations.

In the case of Nero d'Avola, only malic acid shows a statistically significant decrease of 0.07 g/l in EDNA compared to CNA, while the other acids -lactic, citric and tartaric- as well as total acidity, do not show significant changes. In Syrah, only total acidity and lactic acid show a statistically significant reduction after ED treatment.

Regarding the cationic fraction, a significant decrease is observed in both red wines, particularly a reduction of 0.20 g/l in EDNA compared to CNA and 0.15 g/l in EDS compared to CS for RAN, Ca and Cu. For catechins, only in Syrah is a statistically significant increase observed, which is a product of tannin oxidation. In both red wines, TSO₂ shows a statistically significant decrease after tartaric stabilization. Gómez Benítez *et al.* (2003) report that ED reduces the concentration of sulphates more than tartrates. According to Rosária *et al.* (2022), in red wines subjected to membrane filtration, total phenols decreased by between 4.9 and 10.3%.

Regarding color, not statistically significant variations were observed in either wine for both color intensity (IC) and Hue. In the study by Ma *et al.* (2020), IC showed a significant decrease of 35.44% in wines treated with membrane filtration.

One of the main advantages of ED is its sensory neutrality: the main oenological parameters (aroma, color, and taste) remain unchanged, provided that the treatment is carried out within the appropriate operational thresholds (Daufin *et al.*, 2001). Of special interest, however, is the positive decrease observed in the Hue of the filtered wine which means a decrease in the yellow/red propor-

Table 3. Physico-chemical parameters measured in the wine Grillo. The analyses were performed in triplicate for each treatment, and the mean values obtained were compared using Tukey's test at a 95% significance level (Statgraphics centurion, Statpoint Inc., USA, 2005).

Parameter	CG	CFG	EDG
Alcohol (%/vol)	12.95±0.10	12.90±0.10	12.91±0.09 ^{ns}
pH	3.29±0.02 ^a	3.26±0.03 ^a	3.18±0.01 ^b
Total dry extract (g/l)	24.15±2.12	25.40±2.23	22.03±3.00 ^{ns}
Total acidity(g/l)	5.89±0.19	5.97±0.18	5.68±0.32 ^{ns}
Volatile acidity (g/l)	0.34±0.01	0.30±0.05	0.35±0.02 ^{ns}
Malic acid (g/l)	1.27±0.08	1.30±0.14	1.13±0.14 ^{ns}
Lactic acid (g/l)	0.71±0.24	1.10±0.60	0.70±0.63 ^{ns}
Citric acid (g/l)	0.17±0.02	0.12±0.04	0.17±0.03 ^{ns}
Tartaric acid (g/l)	3.06±0.04 ^b	3.17±0.19 ^a	2.60±0.04 ^b
K ⁺ (g/l)	0.69±0.12 ^{ab}	0.73±0.15 ^a	0.40±0.19 ^b
RAN (g/l)	46.00±15.6	48.00±11.3	51.00±1.40 ^{ns}
Ca ²⁺ (mg/l)	66.50±2.12	62.50±4.95	58.00±1.41 ^{ns}
Polyphenols (mg/l)	204±31.4 ^{ab}	226.3±8 ^a	159.7±28.6 ^b
Catechins (mg/l)	17.68±3.14	24.36±4.46	20.86±6.74 ^{ns}
Ashes (g/l)	2.30±0.14 ^a	2.50±0.26 ^a	1.62±0.17 ^b
Cu ²⁺ (mg/l)	0.20±0.01	0.19±0.04	0.13±0.02 ^{ns}
TSO ₂ (mg/l)	87.67±8.39	90.67±10.2	81.33±17.2 ^{ns}
FSO ₂ (mg/l)	30.50±0.71 ^a	31±1.41 ^a	25.50±0.71 ^b

Data reported are mean ± SD; ns, not significant; different letters in the row indicate statistically significant differences.

tion in the wine (Arriagada-Carrazana *et al.*, 2005). The observed reduction in polyphenols after electro dialysis was more pronounced in white wines (−44 mg/L) than in reds, where the effect was negligible. Although ED caused a partial reduction in total polyphenols, this variation is unlikely to produce perceivable sensory changes, as polyphenolic composition is buffered by macromolecules such as tannins and polysaccharides (Vernhet and Moutounet, 2002; Smith *et al.*, 2015). Consequently, the slight polyphenol variation observed after ED treatment can be considered technologically irrelevant under the present industrial conditions. Different types of membranes used in wine filtration have different surface properties that can also influence the extent to which tannins bind to filtration membranes (Vernhet and Moutounet, 2002). According to Smith *et al.* (2015), red wine filtration can be a source of concern for winemakers for two main reasons. Firstly, it can damage the membranes, leading to additional costs related to their replacement and maintenance. Secondly, filtration alters the wine's texture and sensory profile, creating a mouthfeel phenomenon known as "bottle shock", a period of altered sensory characters in the product. The two-way ANOVA highlighted significant interactions between variety and treatment, confirming that the effect of ED is strongly matrix dependent. For white wines (Table 6), significant interactions were found for malic acid ($p < 0.001$), tartaric acid ($p = 0.003$), and free SO₂ ($p = 0.045$). This indicates that the response to ED was not uniform across Catarratto and Grillo: Catarratto showed greater reductions in malic and tartaric acids, while in Grillo the changes were less pronounced, underlining varietal differences in organic acid buffering capacity. For red wines (Table 7), interactions were significant for total acidity ($p < 0.001$), malic acid ($p < 0.001$), Ca²⁺ ($p < 0.001$), and color intensity ($p = 0.021$). These results suggest that ED influenced not only acid balance but also cationic stability and chromatic properties in a way that varied between Nero d'Avola and Syrah. For instance, Syrah exhibited a stronger decrease in total acidity and lactic acid after ED, while Nero d'Avola showed higher sensitivity in Ca²⁺ reduction and associated effects on stability. Overall, these results demonstrate that ED does not exert a uniform effect across varieties but rather interacts with specific compositional characteristics (organic acids, cations, phenolic-color matrix), reinforcing the importance of tailoring stabilization protocols to wine type.

Aromatic component

The results of gas chromatography analyses for the different grape varieties are reported in Tables 8-11, where the area (%) indicates the relative abundance of each compound. Across all wines, both white and red, tangential filtration and tartaric stabilization were found to influence the aromatic composition. In the volatile fraction, eleven compounds were identified, belonging to three main chemical classes: esters, alcohols, and terpenes. Among these, esters represented the most abundant group in all samples.

Among the esters, the main compounds identified were isoamyl acetate, ethyl hexanoate, methyl octanoate, ethyl octanoate, and ethyl decanoate. In general, esters are responsible for fruity and sweet notes, except for ethyl decanoate, which contributes to pungent spicy aromas. As shown in Tables 8 and 9, the concentration of these volatile compounds increased after ED treatment in both white wines.

Regarding alcohols, isoamyl alcohol and 2-phenylethanol were detected. The highest concentration was found for isoamyl alcohol, which imparts pungent and alcoholic notes. Its content was higher in the control samples for both Grillo and Catarratto. This com-

pound subsequently decreased in both wines after ED treatment, dropping from 87.28% in CG to 86.36% in EDG, with a similar trend observed in Catarratto, where the component decreased by 3.02% in EDC. Linalool, geraniol, citronellol, and nerol, which contribute floral and fruity aromas such as rose, citronella, and citrus are among the most relevant terpenes in winemaking. In our study, the terpene components identified were d-limonene and β -linalool, both associated with citrus-floral notes. The highest concentrations were recorded in Catarratto after ED treatment, with d-limonene increasing to 3.48% in EDC compared to 2.64% in CC, and β -linalool increasing from 0.06% in CC to 0.16% in EDC. In Grillo, the values for d-limonene were similar between treatments, 3.72% in CG and 3.70% in EDG, while β -linalool remained unchanged at 0.10% in both cases. Within this wide range of volatile compounds, monoterpene alcohols have the greatest sensory impact. In particular, linalool and geraniol are characterized by very low perception thresholds, 15 μ g/L and 30 μ g/L, respectively (Ferreira *et al.*, 2002). Approximately 50% of the total monoterpenes are found in the grape skin; however, geraniol is mainly associated with the berry skin, while linalool is also evenly distributed within the pulp (Wilson *et al.*, 1986).

For Nero d'Avola variety (Table 10), eleven volatile compounds were identified, while for Syrah (Table 11), ten were detected, as ethyl hexanoate was not present in this case. In Nero d'Avola, esters showed an increase after cross-flow filtration compared to the untreated sample, as shown in Table 8, and then stabilized after electro dialysis, reaching values similar to those of the control sample (CND). Isoamyl acetate recorded values of 0.29 in CND, 0.83 in CFND, and 0.22 in EDND, while ethyl hexanoate showed 3.48 in CND, 0.07 in CFND, and 3.06 in EDND.

A different trend was observed for Syrah, where ester concentrations tended to decrease after ED treatment compared to the control. Among the alcohols, isoamyl alcohol was the most abundant compound in Syrah compared to Nero d'Avola, with values of 83.30 in CS and 0.74 in CND, respectively. This component increased significantly in CFND to 77.43 and then decreased after ED to 0.87. Conversely, in Syrah, this compound decreased in CFS to 67.07 and then increased to 82.24 in EDS. Finally, among terpenes, d-limonene showed lower concentrations after ED in both red wines, while β -linalool increased by 0.04% in Nero d'Avola and by 0.10% in Syrah. Overall, the GC-MS analysis showed that ED modified the volatile composition of wines in a matrix-dependent manner. In white wines (Catarratto, Grillo), increases in compounds such as β -linalool and d-limonene, together with a reduction in isoalcohols, indicate chemical trends potentially associated with fresher fruity-floral notes. In contrast, red wines (Nero d'Avola, Syrah) displayed decreases in some esters (e.g., ethyl butyrate, isoamyl acetate), likely due to phenolic-volatile interactions and smaller pH shifts, suggesting that the impact of ED on aroma depends strongly on wine style. While these changes suggest possible sensory implications, no sensory analysis was performed in this study, and further work is needed to confirm these relationships. Compared to conventional clarification processes, such as centrifugation and sheet filtration, diatomaceous earth filtration, etc., cross-flow filtration can bring the following benefits such as the combination of clarification, microbiological stabilization and sterile filtration in one single continuous highly automated operation, and the elimination of the use of diatomaceous earth. Thereby, reducing production costs and the problem of waste disposal leading to an improvement in work safety and production (El Rayess *et al.*, 2011). The interpretations of the results derive from independently processed sub-batches under real industrial-scale operating conditions. Therefore, the statistically supported differ-

Table 4. Physico-chemical parameters measured in the wine Nero d'Avola. The analyses were performed in triplicate for each treatment, and the mean values obtained were compared using Tukey's test at a 95% significance level (Statgraphics centurion, Statpoint Inc., USA, 2005).

Parameter	CNA	CFNA	EDNA
Alcohol (%/vol)	13.27±0.03 ^{ab}	13.33±0.05 ^a	13.22±0.05 ^b
pH	3.42±0.01 ^a	3.43±0.02 ^a	3.34±0.02 ^b
Total dry extract (g/l)	35.33±0.38 ^a	35.46±0.54 ^a	34.36±0.43 ^b
Total acidity(g/l)	6.21±0.10	6.25±0.09	6.143±0.11 ^{ns}
Volatile acidity (g/l)	0.38±0.02	0.37±0.02	0.38±0.02 ^{ns}
Malic acid (g/l)	0.13±0.01 ^a	0.13±0.01 ^a	0.06±0.01 ^b
Lactic acid (g/l)	1.03±0.09	1.01±0.09	0.97±0.02 ^{ns}
Citric acid (g/l)	0.24±0.01	0.23±0.01	0.22±0.01 ^{ns}
Tartaric acid (g/l)	3.23±0.16	3.24±0.11	2.99±0.14 ^{ns}
K ⁺ (g/l)	1.00±0.06 ^a	1.03±0.09 ^a	0.80±0.06 ^b
RAN (g/l)	77.50±4.95	75.50±0.71	66.0±4.24 ^{ns}
Ca ²⁺ (mg/l)	80.50±0.71	80.00±1.41	77.5±0.71 ^{ns}
Polyphenols (mg/l)	1823±8.08	1819±38.1	1818±34.7 ^{ns}
Catechins (mg/l)	126±1.92	125.40±1.62	129.50±2.47 ^{ns}
Ashes (g/l)	3.11±0.08 ^a	3.15±0.11 ^a	2.69±0.09 ^b
Cu ²⁺ (mg/l)	0.11±0.02 ^{ab}	0.13±0.02 ^a	0.06±0.03 ^b
TSO ₂ (mg/l)	92.33±3.06 ^a	90.33±5.51 ^a	80.67±4.0 ^b
FSO ₂ (mg/l)	32.00±1.41 ^a	33.50±0.71 ^a	27.50±0.71 ^b
Anthocyanins (mg/l)	235±10	225.7±23.9	223.7±20.8 ^{ns}
IC	6.50±3.90	6.40±0.71	6.70±3.28 ^{ns}
Hue	0.69±0.03	0.69±0.03	0.63±0.02 ^{ns}

Data reported are mean ± SD; ns, not significant; different letters in the row indicate statistically significant differences.

Table 5. Physico-chemical parameters measured in the wine Syrah. The analyses were performed in triplicate for each treatment, and the mean values obtained were compared using Tukey's test at a 95% significance level (Statgraphics centurion, Statpoint Inc., USA, 2005).

Parameter	CS	CFS	EDS
Alcohol (%/vol)	13.63±0.12	13.63±0.12	13.52±0.10 ^{ns}
pH	3.60±0.01 ^a	3.59±0.01 ^a	3.51±0.01 ^b
Total dry extract (g/l)	36.41±0.47 ^a	36.05±0.76 ^a	34.51±0.59 ^b
Total acidity(g/l)	5.67±0.02 ^a	5.63±0.06 ^{ab}	5.56±0.05 ^b
Volatile acidity (g/l)	0.64±0.04	0.64±0.04	0.62±0.03 ^{ns}
Malic acid (g/l)	0.03±0.03	0.07±0.03	0.03±0.04 ^{ns}
Lactic acid (g/l)	1.16±0.02 ^a	1.18±0.01 ^a	1.06±0.03 ^b
Citric acid (g/l)	0.14±0.04	0.15±0.05	0.14±0.05 ^{ns}
Tartaric acid (g/l)	2.47±0.11	2.48±0.11	2.34±0.07 ^{ns}
K ⁺ (g/l)	1.14±0.06 ^a	1.14±0.06 ^a	0.99±0.05 ^b
RAN (g/l)	81.7±0.60 ^a	81.33±0.60 ^a	69.33±0.60 ^b
Ca ²⁺ (mg/l)	67.3±0.60 ^b	69.33±0.60 ^a	67.33±0.58 ^b
Polyphenols (mg/l)	2324±95	2290±73	2283±63 ^{ns}
Catechins (mg/l)	177±0.70 ^b	175.70±0.7 ^b	181.7±0.71 ^a
Ashes (g/l)	3.29±0.02 ^a	3.31±0.06 ^a	2.94±0.07 ^b
Cu ²⁺ (mg/l)	0.19±0.01 ^a	0.17±0.03 ^{ab}	0.12±0.04 ^b
TSO ₂ (mg/l)	95±6.20 ^a	89±3.0 ^{ab}	81.67±5.8 ^b
FSO ₂ (mg/l)	25.70±0.60 ^a	24.33±0.6 ^a	21.67±0.58 ^b
Anthocyanins (mg/l)	265±2 ^a	254±5 ^b	238±2 ^c
IC	7.60±3.89	7.50±0.80	7.70±3.76 ^{ns}
Hue	0.68±0.04	0.69±0.03	0.65±0.03 ^{ns}

Data reported are mean ± SD; ns, not significant; different letters in the row indicate statistically significant differences.

ences observed reflect process-level variability rather than analytical repetition. While the conclusions are bounded to the investigated varieties and technological settings, they provide the evidence of treatment performance within an industrial membrane-based stabilization framework.

Table 6. Bidirectional ANOVA for interaction between variety (Catarratto, Grillo) and treatment (C, CF, ED).

Parameter	Variety × Treatment
Alcohol (%/vol)	0.848
pH	0.274
Total dry extract (g/l)	0.115
Total acidity(g/l)	0.895
Volatile acidity (g/l)	0.274
Malic acid (g/l)	0.000*
Lactic acid (g/l)	0.447
Citric acid (g/l)	0.317
Tartaric acid (g/l)	0.003*
K ⁺ (g/l)	0.881
RAN (g/l)	0.710
Ca ²⁺ (mg/l)	0.259
Polyphenols (mg/l)	0.572
Catechins (mg/l)	0.707
Ashes (g/l)	0.652
Cu ²⁺ (mg/l)	0.169
TSO ₂ (mg/l)	0.871
FSO ₂ (mg/l)	0.045*

*p<0.05.

Table 7. Bidirectional ANOVA for interaction between variety (Nero d’Avola, Syrah) and treatment (C, CF, ED).

Parameter	Variety × Treatment
Alcohol (%/vol)	0.696
pH	0.673
Total dry extract (g/l)	0.253
Total acidity(g/l)	0.000*
Volatile acidity (g/l)	0.606
Malic acid (g/l)	0.000*
Lactic acid (g/l)	0.408
Citric acid (g/l)	0.763
Tartaric acid (g/l)	0.550
K ⁺ (g/l)	0.535
RAN (g/l)	0.744
Ca ²⁺ (mg/l)	0.000*
Polyphenols (mg/l)	0.797
Catechins (mg/l)	0.624
Ashes (g/l)	0.381
Cu ²⁺ (mg/l)	0.435
TSO ₂ (mg/l)	0.572
FSO ₂ (mg/l)	0.101
IC	0.021*
Hue	0.115

*p<0.05.

Conclusions

This study evaluated the application of cross-flow filtration and electro dialysis for wine stabilization under real industrial operating conditions, using independently processed sub-batches for each variety × treatment combination. Cross-flow filtration caused slight alterations in wine quality. The subsequent tartaric stabilization process improved the quality of wine. This effect is particularly evident in pH, which showed a significant reduction of about 0.10 in all wines after ED, and more notably in the significant decrease of cationic fractions such as K⁺ and Ca²⁺. An additional relevant aspect concerns the chromatic stability of red wines, which remained substantially unchanged following treatment, confirming the preservation of a key quality parameter. Cross-flow filtration and tartaric stabilization influenced the aromatic profile of the wines; in some cases, electro dialysis appeared to mitigate aromatic variations observed after cross-flow filtration, suggesting a matrix-dependent modulation of volatile composition. Overall, ED influenced volatile composition differently in white and red wines, suggesting matrix-dependent effects driven by pH shifts and phe-

Table 8. Chemical compounds and their relative abundances on wine Catarratto after cross-flow filtration and after ED by gas chromatography.

Compound	CC	Area % CFC	EDC
Isoamyl alcohols	67.84	1.00	64.82
Ethyl butyrate	0.20	76.95	0.11
Isoamyl acetate	0.04	0.34	0.21
Ethyl hexanoate	0.14	1.99	0.73
d-limonene	2.64	0.23	3.48
β-linalool	0.06	7.73	0.16
2 Phenyl ethanol	0.17	17.00	0.11
Methyl octanoate	0.29	0.13	0.37
Ethyl octanoate	1.84	0.10	0.55
Isoamyl hexanoate	0.05	2.21	0.31
Ethyl decanoate	0.06	3.32	0.43

Table 9. Chemical compounds and their relative abundances on wine Grillo after cross-flow filtration and after ED by gas chromatography.

Compound	CG	Area % CFG	EDG
Isoamyl alcohols	87.28	45.00	86.36
Ethyl butyrate	0.02	0.10	0.04
Isoamyl acetate	0.08	0.13	0.07
Ethyl hexanoate	0.13	3.40	0.20
d-limonene	3.72	0.14	3.70
β-linalool	0.10	5.40	0.10
2 Phenyl ethanol	0.04	0.20	0.15
Methyl octanoate	0.06	0.22	0.22
Ethyl octanoate	0.05	0.21	0.21
Isoamyl hexanoate	0.13	0.40	0.06
Ethyl dodecanoate	0.02	0.70	0.10

nolic-volatile interactions. While these chemical trends may have sensory implications, further studies combining GC-MS with sensory evaluation are needed to validate their impact on wine quality. This study confirms that the application of ED does not denature colloids, and has no significant effects on total acidity. Therefore, it represents a safe and effective method for tartaric stabilization. The introduction of ED into the winemaking process represents a paradigm shift in the management of wine tartaric stability, replacing traditional high-energy-impact methods (cold treatments) with more selective and sustainable membrane technologies. The technical effectiveness, sensory neutrality, and operational flexibility of ED are well-established in various production contexts; however, its implementation on an industrial scale requires integrated analysis from multiple perspectives: technical-operational, oenological-qualitative, economic-managerial, and environmental.

The results obtained show that ED effectively reduced pH, K⁺, and tartaric acid, confirming its stabilizing effect; decreased total salt concentration, as indicated by reduced ash content; caused a partial reduction in total polyphenols, which should be assessed on a varietal basis; and maintained alcohol, volatile acidity, minor acids, and Cu²⁺ within acceptable limits. In summary, ED confirms its selective effectiveness and compatibility with wine quality, par-

ticularly for white and rosé wines intended for long-term storage. However, potential polyphenol reduction should be considered in less-structured wines or those intended for ageing. Future studies should integrate sensory panels to confirm consumer-relevant impacts. Despite its numerous advantages, ED still faces certain barriers to large-scale adoption: high initial costs for equipment and membranes; sensitivity to fouling, particularly in wines rich in polysaccharides and phenolic compounds (Nazir *et al.*, 2011); and the need for trained personnel and continuous process monitoring.

Future prospects include the development of anti-fouling selective membranes, integration with real-time sensors and automated control software, and combining applications with other technologies (e.g., flotation, reverse osmosis) to optimize the winemaking process.

Table 10. Chemical compounds and their relative abundances on wine Grillo after cross-flow filtration and after ED by gas chromatography.

Compound	Area %		
	CND	CFND	EDND
Isoamyl alcohols	0.74	77.43	0.87
Ethyl butyrate	53.89	0.04	68.01
Isoamyl acetate	0.29	0.83	0.22
Ethyl hexanoate	3.48	0.07	3.06
d-limonene	7.79	0.10	4.45
β-linalool	0.03	0.02	0.07
2 Phenyl ethanol	0.15	0.08	0.11
Methyl octanoate	0.22	0.02	0.31
Ethyl octanoate	0.08	2.78	0.15
Isoamyl hexanoate	0.15	0.84	0.18
Ethyl decanoate	0.02	0.11	0.06

Table 11. Chemical compounds and their relative abundances on wine Syrah after cross-flow filtration and after ED by gas chromatography.

Compound	Area %		
	CS	CFS	EDS
Isoamyl alcohols	83.30	67.07	82.24
Ethyl butyrate	0.13	4.11	0.04
Isoamyl acetate	0.84	0.07	0.25
d-limonene	0.15	0.11	0.10
β-linalool	0.06	0.42	0.16
2 Phenyl ethanol	0.10	0.29	0.07
Methyl octanoate	0.65	0.05	0.19
Ethyl octanoate	0.22	0.08	0.13
Isoamyl hexanoate	0.92	0.07	0.15
Ethyl decanoate	0.15	0.31	0.07

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