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Fortified or passito sweet wines from Aleatico grapes subjected to different dehydration conditions: chemical and aromatic profile using destructive and non-destructive analyses

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ABSTRACT

Sweet or fortified wines hold significant cultural and economic value, particularly in regions where they are traditionally produced. Aleatico is a distinctive wine grape variety known for its unique aromatic characteristics, particularly used to produce sweet red wine. The drying technique significantly influences the wine's aroma and flavour profile. In the present study, the impact of different dehydration methods on Aleatico sweet and fortified wine quality was studied. Aleatico grapes were subjected to cold room (CR) dehydration (9 °C, 60 % RH) or room temperature (RT) dehydration (21 °C, 45 % RH) until 35 % of weight loss was reached. Dehydrated grapes were then fermented to obtain passito wines (Sweet RT, Sweet CR) or ethanol was added to the must to produce fortified wines (Fortified RT, Fortified CR). Non-destructive techniques (NIR-AOTF and E-nose) were applied for fast monitoring of wine differences together with chemical destructive analyses. At the end of the dehydration process significant increase in sugars and total extract was observed, without a significant difference between the two dehydration methods. Significant differences were observed in the time required to reach the fixed weight loss, with RT taking 19 days and CR only 47 days. Moreover, Sweet wines had a higher concentration of alcohols, esters, terpenes and ketones but lower aldehydes than Fortified wines. Sweet wines also had a higher concentration of organic acids but a greater susceptibility to oxidation, despite a higher and chemically more uniform phenolic fraction. E-nose and NIR-AOTF were able to discriminate between Sweet and Fortified wines. However, E-nose present evident limitations in discriminating between different dehydration conditions.

KEYWORDS: wine grape dehydration; sweet wines; fortified wine; electronic nose; NIR-AOTF; VOCs, HPLC phenolic compounds

INTRODUCTION

Post-harvest grape water loss when producing wine can have different terms: dehydration, drying, and withering (Mencarelli & Tonutti, 2013). This process can be carried out in different ways in post-harvest as well as on vines. The environmental condition where the dehydration takes place significantly affects the composition of the grapes (Bianchi *et al.*, 2024; Figueiredo-González *et al.*, 2013a; Sanmartin *et al.*, 2021; Santini *et al.*, 2023).

The choice of the drying system is often related to local traditions, climatic conditions, or the preference of the producer and their winemaking objectives. Dehydration in a cold room allows for controlling temperature and relative humidity more accurately (Corona *et al.*, 2020; Urcan *et al.*, 2017; Zenoni *et al.*, 2020).

In an open field environment, ambient conditions cannot be controlled to proceed with grape water loss whereas, in a close ambient, temperature, relative humidity, and airflow can be managed. Managing these parameters allows one to model the kinetics of water loss and modulate this process. Temperature is one of the main factors affecting the quality of postharvest dehydrated grapes (Chen *et al.*, 2021; Cirilli *et al.*, 2012; Nicoletti *et al.*, 2013; Shmulevitz *et al.*, 2023). Maintaining optimal and constant temperature reduces fungi formation (Negri *et al.*, 2017; Stefanini *et al.*, 2016), avoid berry's internal fermentations and oxidative phenomena with consequent aromatic loss (Bianchi *et al.*, 2023; Niewierowski *et al.*, 2021; Petriccione *et al.*, 2018; Scalzini *et al.*, 2024).

By controlling the dehydration parameter, especially temperature, we can modulate the different metabolisms, such as sugar accumulation, polyphenol synthesis, organic acid degradation, and volatile organic compound formation, occurring in the berry during postharvest water loss, can be modulated (Bianchi *et al.*, 2024; Bonghi *et al.*, 2012; Mencarelli & Bellincontro, 2013; Niewierowski *et al.*, 2021). The possibility of managing water loss kinetics also allows for predicting the duration of the process, and consequently the different metabolism kinetics: reaching 30 % weight loss in a shorter or longer time makes a significant difference. Additionally, the amount of water loss is also crucial for the final quality (Mencarelli & Tonutti, 2013). Different metabolisms are activated depending on the speed of the process; for instance, a slow dehydration rate favours polyphenol metabolism, leading to an accumulation of tannins and anthocyanins, while a rapid water loss might accelerate respiratory metabolism, increasing oxidative stress and altering the final composition of the berry (Mencarelli & Bellincontro, 2020; Modesti *et al.*, 2021; Santini *et al.*, 2023; Shmulevitz *et al.*, 2023; Urcan *et al.*, 2017).

Dehydration without control of ambient parameters, at room temperature, on the other hand, poses different challenges and consequences related to uncontrolled variations, therefore fluctuating temperatures and humidity which can lead to a less uniform process. In such conditions, the risks of contamination by mould, bacteria, or pathogens, which may alter the aromatic

profile of the berries, are higher. Contamination can induce metabolic processes and unwanted enzymatic activities, such as polyphenol oxidation carried out by polyphenol oxidase, increasing activity of lipoxygenase, the hydrolysis of glycosidic compounds carried out by β -glucosidase which influences the aromatic profile, and other cell wall enzymes involvement (Antelmi *et al.*, 2010; Bellincontro *et al.*, 2017; Botondi *et al.*, 2011; Chkaiban *et al.*, 2007).

Once the grapes are dehydrated, fermentation starts. However, sweet wine fermentation involves several challenges compared to dry wine fermentation. Hence, fermenting high-sugar musts can lead to elevated volatile acidity and extended fermentation times. One of the main risky phenomena is fermentation arrest due to the high sugar concentration. However, to produce fortified wines, fermentation needs to stop once a certain level of alcohol or residual sugar has been reached. The most common techniques to block fermentation are the addition of sulfur dioxide or by lowering the temperature. In the case of fortified wine, a common practice is to add grapes-derived alcohol during fermentation or directly to the must before fermentation starts. This technique is particularly diffuse in Spain and Portugal (Ingrassia *et al.*, 2018; Ossola *et al.*, 2017; Reboredo-Rodríguez *et al.*, 2015).

The addition of ethanol increases the wine body, inhibits microorganism growth, modifies significantly the wine aroma and, above all, protects the wine against the oxidative process (Jasmins *et al.*, 2023; Pereira *et al.*, 2019; Verzera *et al.*, 2021). Unfortunately, the addition of alcohol impacts significantly the aroma of wine and the ethanol smell can be excessively dominant. For this reason, but not only, long aging is an important process of the most famous fortified wines such as Pedro Ximenez and Porto (Reboredo-Rodríguez *et al.*, 2015). Ageing carried out in wood barrels is an oxidative process that first enables ethanol oxidation, mitigating its odour and flavour, and subsequently leads to changes in other oxygen-susceptible compounds (Bianchi *et al.*, 2025b; Reboredo-Rodríguez *et al.*, 2015). To be effective, the ageing in the barrel must be very long, *i.e.*, years and years. Passito sweet wines, on the other hand, without alcoholisation, in the same ageing condition, undergo different biochemical changes. In fact, they are more susceptible to oxidative phenomena, to sugar polymerisation with a consequent decrease in taste perception in the mouth (Abreu *et al.*, 2021; Guerrero-Chanivet *et al.*, 2024; Tredoux & Silva Ferreira, 2012).

Behind a sweet wine, there are multiple factors and challenges that influence the final quality. As such, monitoring the chemical changes during dehydration, production, and ageing can be crucial for maintaining wine quality. In the last decade, the E-nose and NIR methods have emerged as useful tools to rapidly monitor grape and wine quality (Littarru *et al.*, 2025). For instance, E-nose has been effectively used to identify the aromatic profiles of wines produced with different production methods, from different varieties or different geographical origins (Alfieri *et al.*, 2024; Gonzalez Viejo & Fuentes, 2022; Lozano *et al.*, 2016; Tari *et al.*, 2024). Moreover, the NIR-AOTF approach, if properly combined

with chemometric approaches, can be used for rapid and non-destructive identification of chemical composition (Dos Santos Costa *et al.*, 2019; Genisheva *et al.*, 2018; Littarru *et al.*, 2025; Rouxinol *et al.*, 2022; Yu *et al.*, 2018).

In this context, this study aimed to monitor the effect of dehydration on the production of sweet and fortified Aleatico wines, with a specific focus on three key hypotheses. First, it investigated the advantages of using low temperatures for grape dehydration. Second, it examined whether alcoholisation significantly alters the aroma of wine compared to passito sweet wine. Third, it explored the potential of non-destructive techniques in differentiating the different wines.

MATERIALS AND METHODS

1. Equipment and winemaking process

Aleatico grape bunches (*Vitis vinifera* L.) were manually harvested (240 ± 3 g/L of sugar) in 2022 at Famiglia Cotarella-Falesco in Montecchio (TR), Italy and then were placed in perforated crates (about 6 kg grape each crate).

The grapes (6 crates for each sample) were subjected to dehydration: i) at room temperature (RT) of 21 ± 2 °C and 45 ± 5 % RH (relative humidity) with an airflow of 1 m/s, or ii) in a cold room (CR) at 9 ± 2 °C, 60 ± 5 % RH (maintained using a dehumidifier) with an airflow of 1 m/s. Temperature and RH sensors were used for daily monitoring developed by DIBAF (University of Tuscia, Viterbo) (Santini *et al.*, 2023); airflow was measured by using an anemometer (Lutron LM-8000, Coopersburg, PA, USA). The 6 crates were weighed each day and dehydration was stopped at approximately 35 % weight loss when the grape reached about 400 g/L of total sugars. Dehydrated grapes from RT or CR were poured into a crusher–destemmer and each must divided into two lots: the first lot was immediately fermented to produce sweet wine (Sweet RT and Sweet CR); The other was used to produce fortified wines (Fortified RT and Fortified CR).

To produce fortified wines from RT or CR, each must was roughly filtered (metal net) to eliminate debris and divided into three 5 L glass jars for each sample. Each jar had 95 % (v/v) of alcohol (food grade) added until an alcohol content of 15 % (v/v) was reached; after one month, the wines were bottled in 0.75 L glass bottles with a silicon stopper and stored at 15 °C.

To produce passito sweet wines, must from dehydrated grapes at RT or CR, without filtration, were divided into three 5 glass jars, and 8 g/hL of $K_2S_2O_5$ and 15 g/hL of Zymaflore X5 yeast (Laffort srl, Tortona (AL), Italy) added to each one. At day 3 from the start of fermentation, 20 g/hL of fermentation activator (Nutriferm® Vit Flo, Enartis srl, San Martino (NO), Italy) were added. Pigeage was performed every day. The fermentation process was blocked adding 12 g/hL of $K_2S_2O_5$ at a must sugar concentration of around 160–180 g/L. Then, the four wines (Fortified RT, Fortified CR, Sweet RT and Sweet CR) were bottled in 0.75 L glass bottles with silicon stopper and stored at 15 °C. No malolactic fermentation was performed.

2. Chemical analyses

Grapes were analysed at harvest and the end of the dehydration process (3 samples of 50 berries from different crates). Wine analysis was performed on finished wines on 3 bottles for each sample. Chemical determinations were carried out by a calibrated Fourier transform infrared WineScan™ FT 120 (Foss Analytics, Hillerød, Denmark) to determine: sugars (g/L hexoses), pH, titratable acidity (tartaric acid g/L), volatile acidity (g/L acetic acid), malic acid (g/L), tartaric acid (g/L), citric acid (g/L), gluconic acid (g/L), ash (g/L), YAN (g/L), total extract (g/L), glycerol (g/L), total anthocyanins (mg/L malvidin) and total polyphenols (mg/L gallic acid). The accuracy of the WineScan™ analyses was validated, periodically, by destructive analyses performed using the reference OIV methods as reported by Bianchi *et al.* (2021).

The chromatic characteristics were detected by a Benchtop CLM-196 colorimeter (Eoptis-38121 Trento (TN)-ITALY). The colour values were expressed using the native coordinates CIE $L^*a^*b^*$ and the cylindrical coordinates, h^* and C^* , calculated according to Modesti *et al.* (2021). The colour differences among samples (ΔE^*_{ab}) were also calculated as previously reported (Bianchi *et al.*, 2025a) and are expressed in CIELAB units.

3. VOC analysis

Volatile organic compound (VOC) analyses were carried out as previously reported (Mastrangelo *et al.*, 2023). In particular, 10 mL of wine sample and 100 µL of a 2-octanol solution at 500 mg/L was added as an internal standard. The sample was deposited on a Hypersep Retain Pep (Thermo Fisher Scientific, Milan, Italy) cartridge (60 mg), activated with 2 mL dichloromethane, 2 mL methanol and 2 mL water. The analytes were eluted with 5mL of dichloromethane, collected in soviel on the bottom of which 2-grams of anhydrous sodium sulfate had been inserted and placed in the freezer overnight. Prior to GC-MS analysis, samples were filtered with a cellulose filter to remove sodium sulfate and concentrated to a final volume of 200 µL under a stream of N_2 .

The GC apparatus consisted of a Trace GC ultra-gas chromatograph with a Trace DSQ with quadrupole mass detector (Thermo Fisher Scientific, Milan, Italy) and a Stabilwax DA capillary column (Restek, Bellefonte, PA, USA; 30 m, 0.25 mm *i.d.*, and 0.25 µm film thickness). The carrier gas was He with a constant flow of 1.0 mL/min.

GC temperature ramp was programmed as follows: from 45 °C (maintained for 1-minute) to 100 °C (maintained for 1-minute) at 3 °C/min, then to 240 °C (maintained for 10 minutes) at 5 °C/min. The injection was performed at 250 °C in splitless mode and the volume injected is 1 µL.

The identification of compounds was carried out as previously reported (Mastrangelo *et al.*, 2023), following a triple criterion: (i) by comparing compound mass spectra and retention time with those of pure standards, (ii) matching their respective mass spectra with those present in online libraries Willey and NIST 08, (iii) by comparing linear retention index

(LRI) calculated under our analytical conditions with already published LRI calculated on polar columns.

Quantification was carried out by normalisation of integrated areas in total ion current with the area of 2-octanol (internal standard). Analyses were performed in triplicate and GC-MS parameters were obtained by using Xcalibur software (version 4.1, Thermo Fisher Scientific, Milan, Italy).

4. HPLC analysis

The comprehensive analysis of polyphenols within grape and wine samples involved high-performance liquid chromatography (HPLC) using the method previously reported (Tari *et al.*, 2024).

1 mL of grape juice or wine, diluted at a 1:1 ratio with phase A, was extracted from the samples. The obtained sample was filtered through a 0.45 μ m PVDF (Polyvinylidene fluoride) filter before being injected into the HPLC system (Tari *et al.*, 2024): PU-2089 Plus quaternary pump (Jasco International Co., Ltd., Tokyo, Japan), equipped with a degasser, an AS-2057 Plus autosampler (Jasco International Co., Ltd., Tokyo, Japan), and a CO-2060 Plus column oven (Jasco International Co., Ltd., Tokyo, Japan). Detection was conducted using a UV-2070 Plus visible detector (Jasco International Co., Ltd., Tokyo, Japan). The acquired data underwent processing via ChromNAV (software version 2.3). The separation took place using a DionexAcclaim®120 C18 column, 5 μ m, 4.6 $\times$ 250 mm (Thermo Fisher Scientific, Milan, Italy), maintained at a constant temperature of 40 °C, with a mobile phase flow rate set at 0.5 mL/min. The mobile phase comprised a ternary gradient: solvent A = 50 mM ammonium dihydrogen phosphate adjusted to pH 2.6 with phosphoric acid, solvent B = 20 % solvent A and 80 % acetonitrile, and solvent C = 0.2 M orthophosphoric acid adjusted to pH 1.5 with NaOH and the solvent gradient used as reported in Pettinelli *et al.* (2024).

For the qualitative and quantitative analysis of individual polyphenols in the studied matrix, the construction of calibration curves for phenolic compounds of primary interest was performed. Given the extensive variety of polyphenols present in grape and wine matrices, a total of 33 standards (Sigma-Aldrich, Steinheim, Germany) were thoughtfully selected for this purpose.

5. E-Nose analysis

The electronic nose (E-nose) employed in this study was designed and assembled at the University of Rome Tor Vergata, incorporating eight quartz microbalances (QMB). The functionalisation scheme of the metalloporphyrin defining the pattern of the eight sensors is as follows: QMB1: Mn-TPP (tetraporphin), QMB2: Co-p-OCH₃-TPP, QMB3: Sn-TPP, QMB4: Rh-TPP, QMB5: Co-p-TPP, QMB6: Cr-TPP, QMB7: Co-TPP, QMB8: Ru-TPP (Mastrangelo *et al.*, 2023; Saevels *et al.*, 2004). These sensitive molecules, synthesised at the Department of Chemical Sciences and Technologies of the University of Rome Tor Vergata, were specifically chosen for their proven sensitivity to VOCs. In these sensors,

minute changes in mass (Δm) on the absorbing layer of the quartz surface induce a frequency shift (Δf) in the electrical output signal of the oscillator circuit. Within a range of minor alterations, the change in frequency (Δf) is directly proportional to the change in mass (Δm). The variation in the oscillation frequency of quartz microbalances occurs upon the adsorption of volatile molecules from the sample's gaseous phase. Each sensor is linked to a 3-point oscillator, and the output signal relies on the resonant frequency of the microbalance. The instrument is powered through a micro USB connector, also serving for data transmission. Frequency measurement is executed by a calibrated counter with a reference resonator, boasting a frequency resolution on the order of 0.1 Hz. The mass measurement resolution is 0.17 ng. Before commencing the analysis, the initial step involved a nose-cleaning phase by connecting the tubes to a filter containing calcium chloride for over an hour until stability signs were evident on the scales. The analyses were conducted on 10 mL of wine, incubated in closed 25 mL vials (fitted with silicone septum) at 30 °C for 20-minutes. The equilibrated headspace was then extracted for 20-minutes using a stream of filtered air and directed into the electronic nose sensor cell. Each sampling unit comprised 3 measurements. The assessment of aromatic enrichment in the headspaces involved evaluating the signal of the oscillation frequency of individual sensors. This signal was compared to a null measurement reference, which consisted of air measurements devoid of odorous molecules and humidity. The latter served as a flow of pure air, cleaning the electronic nose for 20-minutes and establishing the reference signal. The ensemble of sensor signals formed distinct models (fingerprints) encoding the global composition of the headspace.

6. NIR-AOTF spectroscopy analysis

The objective of the analysis using the NIR-AOTF spectrophotometer (Luminar 5030 Miniature Hand-held AOTF-NIR Analyzer, Brimrose, Baltimore, MD) was to assess the discriminative capacity of the acquired spectra in identifying significant differences among wine samples (Antolini *et al.*, 2021). The operational procedure involved placing the wine inside a test tube, directly in contact with the external probe of the instrument. Measurements were conducted in specular reflectance (R), and the “raw” spectra were acquired in transmittance (T). The spectral acquisition spanned the range of 1100–2600 nm, with a measurement step of 2 nm, and an acquisition speed of 10 scans/s was employed. A total of 20 spectra were acquired. Subsequently, the measured spectra were transformed into absorbance values ($\log 1/T$). Each sample underwent the acquisition of 9 spectra, which were then averaged to obtain 3 representative spectra for each sample thesis.

Furthermore, the working spectral range (1100–2600 nm) encompasses key regions associated with molecular vibrations and electronic transitions, giving information about the composition and characteristics of the wine samples. The adoption of 10 scans/s for acquisition ensures a

high level of data precision, contributing to the reliability of the analysis (Damberg *et al.*, 2015; Páscoa *et al.*, 2020). The conversion of spectra into absorbance values enhances the interpretability of the data, facilitating the identification of distinctive features and patterns within the samples.

7. Statistical analysis

The data analyses were conducted using the JMP Pro 17 software package from SAS Institute located in Cary, NC, USA. In assessing the statistical significance of the experimental data, each sample underwent triplicate analysis. The determination of differences among samples was performed through One Way Completely Randomized ANOVA using CoStat, a software tool from Cohort (version 6.451, Pacific Grove, CA, USA), coupled with Tukey's HSD multiple mean comparison test ($p \leq 0.05$).

The numerical data of NIR and E-nose, once the acquisitions were completed, were assembled within a matrix for subsequent multivariate statistical reprocessing to obtain a discriminative evaluation of non-qualitative supervision (pattern recognition). This approach was pursued on pre-treated data with a normalisation filter (autoscaling) and applying partial least square discriminant analysis (PLS-DA) using leave one out as a validation method.

These analyses and graphical representations were conducted using Matlab R2021a software (Mathworks), supplemented by the PLS Toolbox tool from Eigenvector Research.

RESULTS AND DISCUSSION

1. Effect of grape dehydration in different conditions

A significant difference in dehydration time was observed between RT and CR (Figure 1). At RT, a sugar concentration

of approximately 400 g/L was reached in 19 days, whereas the same concentration required 47 days in CR (Table 1). Despite the different dehydration times, both methods resulted in a weight loss of about 40 %. Moreover, the R^2 values of the regression lines were very high, indicating a strong correlation.

At the end of the dehydration process, as expected, a significant increase in sugars and total extract compared to the initial sample was observed, without a significant difference between the two dehydrated samples (Table 1). Increases in pH and titratable acidity were also observed, especially in the CR sample. This is a typical behaviour of grape dehydration where pH and titratable acidity rise together. This is due to the buffer effect as a consequence of cell wall disruption with the release of cations that produce salts with weak acids such as tartaric and malic acids (Bellincontro *et al.*, 2016; Zoccatelli *et al.*, 2013).

Volatile acidity increased significantly due to the physiological formation of acetic acid as well as the increase of glycerol due to the glyceropyruvic fermentation in the berry under water stress conditions (Bellincontro *et al.*, 2016; Pettinelli *et al.*, 2022). Similarly, gluconic acid increased due to glucose oxidation. Differences were observed for total anthocyanins as well as a significant increase of polyphenols was observed in dehydrated grapes, regardless of the condition (Table 1). This increase can be probably attributable to the concentration effect but also the triggering of the water stress on the phenylpropanoid pathways (Bellincontro *et al.*, 2016; Centioni *et al.*, 2014; Zoccatelli *et al.*, 2013).

After dehydration, a concentration effect was observed for most of the individual phenols identified (Table 2). Comparing a fast dehydration rate at room temperature (RT) with a slow dehydration rate at cold temperature (CR), a significantly higher concentration of anthocyanins, flavonols, flavanols, and two hydroxybenzoic acids is evident in CR grapes.

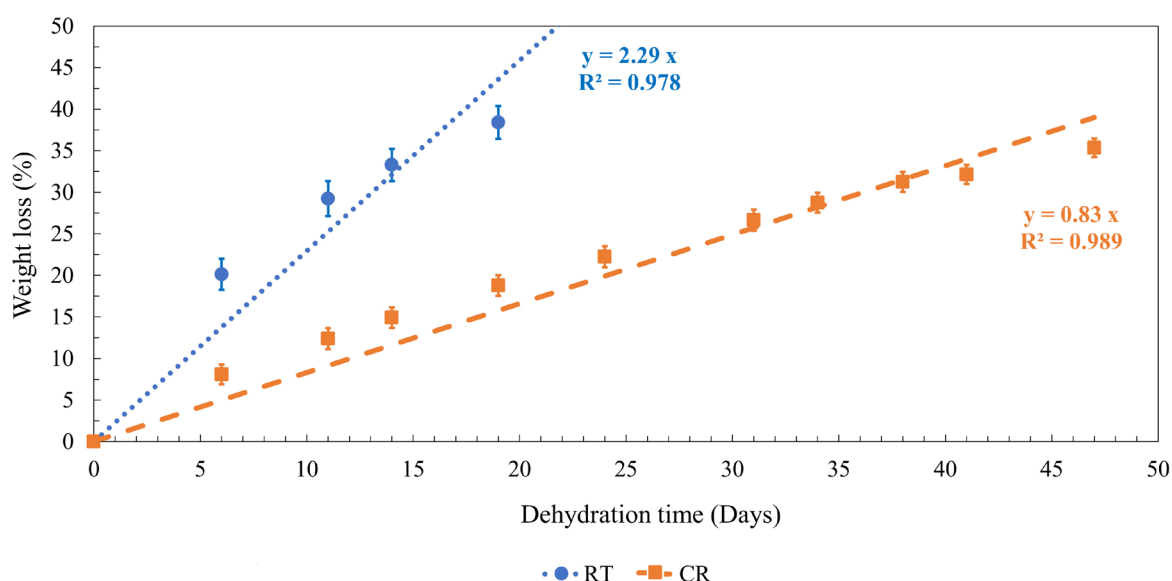


FIGURE 1. Regression line of the weight loss (%) during the dehydration period (days). Data are the mean of 3 crates (Bars indicate the standard deviation). RT = Room temperature; CR = Cold room.

TABLE 1. Chemical parameters of Aleatico grapes at harvest and the end of the dehydration process in the RT and CR conditions.

Parameters	Units	Aleatico grape harvest	RT (19 days)	CR (47 days)
Sugars	g/L hexoses	240 ± 3 b	402 ± 7 a	393 ± 10 a
pH		3.65 ± 0.03 b	3.96 ± 0.04 a	3.89 ± 0.04 a
Titrateable acidity	g/L tartaric acid	4.32 ± 0.17 c	4.73 ± 0.10 b	5.35 ± 0.09 a
Volatile acidity	g/L acetic acid	0.09 ± 0.03 c	0.61 ± 0.03 a	0.51 ± 0.05 b
Malic acid	g/L	1.18 ± 0.08 a	1.07 ± 0.10 a	1.01 ± 0.10 a
Tartaric acid	g/L	2.77 ± 0.04 a	2.16 ± 0.14 b	2.37 ± 0.12 b
Citric acid	g/L	0.24 ± 0.03 a	0.06 ± 0.04 b	0.17 ± 0.04 a
Gluconic acid	g/L	<i>n.d.</i>	2.64 ± 0.12 a	2.09 ± 0.13 b
Ash	g/L	2.07 ± 0.03 b	2.65 ± 0.04 a	2.57 ± 0.06 a
YAN	mg/L	42 ± 1 b	81 ± 7 a	91 ± 7 a
Total extract	g/L	260 ± 8 b	422 ± 15 a	413 ± 21 a
Glycerol	g/L	<i>n.d.</i>	0.82 ± 0.13 a	0.13 ± 0.08 b
Total anthocyanins	mg/L malvidin	229 ± 7 c	299 ± 12 b	337 ± 20 a
Total polyphenols	mg/L gallic acid	1995 ± 34 c	2562 ± 65 b	2851 ± 67 a

Data are the means (± standard deviation) of 3 samples of 50 berries each from different crates using 3 WineScan™ analyses. Different letters in each row indicate statistical differences ($p \leq 0.05$). *n.d.* = not detected.

By calculating the 40 % weight loss based on the initial grape weight to roughly estimate the concentration effect, different trends are observed for anthocyanins. In CR, the final anthocyanin concentration corresponds to the expected value, suggesting a concentration effect due to water loss. Conversely, in RT, the final anthocyanin concentration is lower than expected, indicating a loss of anthocyanins during dehydration rather than a mere concentration effect. In the other classes, flavonols were concentrated in both samples, while flavanols and the two hydroxybenzoic acids showed values significantly higher than expected, particularly in the CR sample. This result confirms the anabolic effect already reported in the literature (Bellincontro & Mencarelli, 2022). In the case of anthocyanins, only a concentration effect was observed in the CR sample (not synthesis, as there was an increase in flavonols, and it is known that the synthesis of these compounds is competitive). In contrast, in the RT sample, the loss of anthocyanins likely occurred due to an oxidation process at room temperature, as anthocyanins are sensitive to oxidation (Bianchi *et al.*, 2022; De Rosso *et al.*, 2010; Figueiredo-González *et al.*, 2013b; Toffali *et al.*, 2011).

2. Chemical characterisation of Sweet and Fortified wines

In Table 3, the main oenological parameters are detailed with their respective significant differences for the wines produced by the four types of grapes.

One of the most obvious results is a higher alcohol concentration in Fortified wines, a result of the addition of ethanol as well as a higher concentration of sugars, as the alcoholic fermentation process was not carried out.

The higher pH (and the lower titrateable acidity) in Fortified wines can be explained by the alcoholisation process without fermentation. It is well known (Chidi *et al.*, 2018; Thoukis *et al.*, 1965) that fermentation acidifies the wine because of the formation of non-volatile acids. As expected, alcoholic fermentation favoured a greater concentration of volatile acidity, due to the high sugar concentration of grapes as well as yeast metabolism. The Fortified wines cannot exhibit this increase, as the addition of alcohol has a sterilising effect, preventing further changes. Additionally, there is no physiological formation of compounds in yeast during fermentation that would contribute to this increase. Among acids, the citric acid showed a significantly higher concentration (4–5 folds) in Sweet wines (Table 3). This behaviour could indicate that during fermentation, the yeasts begin the Krebs cycle thanks to a higher concentration of acetic acid which provides the acetyl group for the formation of citric acid, which probably accumulates because of the difficulty of the Krebs cycle in moving forward (Mendes Ferreira & Mendes-Faia, 2020). Indeed aconitase, the enzyme which transforms citrate into isocitrate, is very sensitive to oxidation; in mammalian cells, it is the enzyme in the Krebs cycle that decreases during aging (Yarian *et al.*, 2006). Grape cells under the dehydration process not only are water stressed but also senesced (D'Onofrio *et al.*, 2019; Zenoni *et al.*, 2016).

The total extract was significantly higher in the Fortified wines due to the higher sugar concentration. The anthocyanins and polyphenols had a significantly lower concentration in the Fortified wines (Table 3). The initial formation of anthocyanin–pyruvic acid adducts has been seen concurrent with the degradation of anthocyanidin monoglucosides in ageing Port (Mateus & de Freitas, 2001),

TABLE 2. Single phenolic compounds (mg/L) in Aleatico grapes at harvest and the end of the dehydration process in the RT and CR conditions.

Phenolic compounds (mg/L)	Aleatico grape at harvest	RT	CR
Cyanidin-3-O-glucoside	3.23 ± 0.71 b	4.23 ± 0.84 b	5.94 ± 0.34 a
Delphinidin-3-O-glucoside	11.25 ± 0.08 b	11.25 ± 0.87 b	13.25 ± 0.18 a
Peonidin-3-O-glucoside	0.20 ± 0.04 b	0.26 ± 0.04 b	0.42 ± 0.04 a
Malvidin-3-O-glucoside	99.19 ± 8.01 c	116.16 ± 3.47 b	155.54 ± 2.89 a
Petunidin-3-O-glucoside	15.97 ± 0.51 c	14.81 ± 0.31 b	17.97 ± 0.51 a
Total Anthocyanins	129.84 ± 1.87 c	146.71 ± 0.97 b	193.12 ± 0.97 a
Myricetin	0.06 ± 0.01 b	0.06 ± 0.02 b	0.16 ± 0.03 a
Quercetin	<i>n.d.</i>	<i>n.d.</i>	1.12 ± 0.04 a
Quercetin-3-O-glucoside	10.77 ± 0.31 c	16.35 ± 0.19 b	16.57 ± 0.94 a
Quercetin-3-O-rutinoside	0.80 ± 0.11 c	2.07 ± 0.29 a	1.20 ± 0.12 b
Quercetin 3-O-ramnoside	<i>n.d.</i>	<i>n.d.</i>	2.22 ± 0.21 a
Kaempferol	1.96 ± 0.13 a	1.12 ± 0.14 b	2.05 ± 0.04 a
Total Flavonols	13.59 ± 0.14 c	19.61 ± 0.35 b	23.21 ± 0.24 a
Catechin	36.46 ± 2.13 a	29.46 ± 1.13 b	39.46 ± 2.13 a
(-)-Epicatechin	111.62 ± 6.55 b	80.50 ± 9.55 c	135.46 ± 7.55 a
Procyanidin B1	<i>n.d.</i>	32.46 ± 1.28 b	53.49 ± 3.05 a
Procyanidin B2	<i>n.d.</i>	120.70 ± 3.50 b	139.46 ± 4.57 a
Procyanidin B3	3.44 ± 0.46 b	2.49 ± 0.34 c	5.12 ± 0.24 a
Total Flavonols	151.53 ± 3.05 c	265.629 ± 3.75 b	372.69 ± 4.05 a
Protocatechic acid	1.52 ± 0.06 c	3.19 ± 0.18 b	4.39 ± 0.16 a
Gallic acid	2.22 ± 0.19 c	6.46 ± 0.59 b	11.31 ± 0.29 a
Total Hydroxybenzoic acids	3.74 ± 0.13 c	9.65 ± 0.45 b	15.70 ± 0.15 a

Data are the means (± standard deviation) of 3 samples of 50 berries each from different crates. Different letters in each row indicate statistical differences ($p \leq 0.05$). *n.d.* = not detected.

but Ossola *et al.* (2017) found that Fortified wines from dehydrated grapes were chromatically unsatisfactory due to the low content of total anthocyanins. Anyway, the fortification does not permit the extraction of chemical compounds which occurs during fermentation. Thus, the effect of postharvest water stress on grape berries is very critical for the colour of Fortified wines, much more than in Sweet wines (Table 3). The CIELAB colour data confirm that sweet wines have lower lightness (L^*) values, indicating a significantly reduced white component and, consequently, a darker colour than fortified wines. This may be attributed to the dilution effect caused by the addition of alcohol, as observed for all the analysed chemical components. Regarding the a^* component (red), sweet wines exhibit significantly higher values compared to fortified wines, whereas the b^* component is lower, indicating a greater presence of the blue component (Arcari *et al.*, 2013). As a result, sweet wines display a redder hue than fortified wines. Furthermore, considering the two grape drying techniques, wines produced from grapes dried in a cold room (CR) exhibit a more intense colour than those obtained from grapes dried at room temperature (RT).

The discrepancy between Sweet and Fortified wines was confirmed by the colour difference value (ΔE^*_{ab}) (Table 4). The results indicate that Sweet RT and Sweet CR exhibit

markedly different colours compared to Fortified RT and Fortified CR, with an ΔE^*_{ab} value greater than 7.00. Conversely, a perceptible difference in the human eye ($\Delta E^*_{ab} = 2.84$) was observed between Sweet RT and Sweet CR. However, this was not the case for the Fortified RT and Fortified CR, which showed a lower colour difference (2.06), indicating no perceptible difference to the human eye (Bianchi *et al.*, 2025a; Martínez *et al.*, 2001).

These data were confirmed by HPLC analyses (Table 5) where we observed the almost disappearance of non-acylated anthocyanins in Fortified wines. Moreover, the other class of polyphenols diminished significantly in Fortified wines. Dehydration at cold temperatures maintained a higher concentration of the different classes, both in Sweet and Fortified wines. The reason for these higher values for wines from cooled, dehydrated grapes is due to a higher concentration measured in grapes after dehydration at low temperatures. As for the significantly lower content in Fortified wines, this is due to the lack of extraction from the solids, as a filtration step was applied. In contrast, extraction occurred during fermentation for the other wines.

TABLE 3. Chemical parameters of wines.

Parameters	Units	Sweet RT	Sweet CR	Fortified RT	Fortified CR
Alcohol	% V/V	12.04 ± 0.04 b	11.84 ± 0.04 c	15.14 ± 0.05 a	15.03 ± 0.07 a
Sugars	g/L hexoses	187 ± 4 b	164 ± 5 c	277 ± 7 a	280 ± 6 a
pH		3.48 ± 0.02 c	3.42 ± 0.01 d	4.10 ± 0.02 a	3.83 ± 0.02 b
Titrateable acidity	g/L tartaric acid	5.92 ± 0.07 a	5.99 ± 0.11 a	4.32 ± 0.14 c	4.91 ± 0.10 b
Volatile acidity	g/L acetic acid	1.11 ± 0.03 a	1.06 ± 0.02 a	0.64 ± 0.05 b	0.69 ± 0.03 b
Malic acid	g/L	1.09 ± 0.05 a	1.07 ± 0.06 a	0.72 ± 0.06 b	0.78 ± 0.04 b
Lactic acid	g/L	0.07 ± 0.04 a	0.11 ± 0.03 a	0.12 ± 0.04 a	0.07 ± 0.04 a
Tartaric acid	g/L	2.18 ± 0.07 a	2.11 ± 0.06 a	1.29 ± 0.04 b	1.35 ± 0.02 b
Citric acid	g/L	1.06 ± 0.02 b	1.24 ± 0.03 a	0.24 ± 0.04 d	0.36 ± 0.01 c
Glycerol	g/L	8.06 ± 0.05 b	8.24 ± 0.07 a	0.28 ± 0.04 b	0.24 ± 0.04 b
Ash	g/L	2.04 ± 0.06 b	2.24 ± 0.04 a	1.64 ± 0.03 c	1.33 ± 0.05 d
YAN	mg/L	15 ± 1 a	16 ± 2 a	8 ± 1 b	9 ± 1 b
Total extract	mg/L	208 ± 6 a	191 ± 6 b	358 ± 12 c	355 ± 22 c
Total anthocyanins	mg/L malvidin	189 ± 3 b	244 ± 7 a	75 ± 8 c	87 ± 9 c
Total polyphenols	mg/L gallic acid	1655 ± 8 b	1765 ± 10 a	635 ± 12 d	695 ± 22 c
Luminosity (L*)		29.53 ± 0.31 c	27.40 ± 0.17 d	34.41 ± 0.26 b	35.75 ± 0.25 a
a*		39.26 ± 0.87 a	38.07 ± 0.22 b	36.65 ± 0.74 c	36.14 ± 0.26 c
b*		14.67 ± 0.20 c	13.22 ± 0.14 d	19.03 ± 0.60 a	17.55 ± 0.09 b
Chroma (C*)		41.91 ± 0.89 a	40.30 ± 0.26 bc	41.30 ± 0.94 ab	40.18 ± 0.27 c
Hue (h*)		20.49 ± 0.13 c	19.15 ± 0.10 d	27.44 ± 0.25 a	25.90 ± 0.06 b

Data are the means (± standard deviation) of 3 wine reps using 3 WineScan™ analyses. Different letters in each row indicate statistical differences ($p \leq 0.05$).

TABLE 4. Colour difference (ΔE^*_{ab}) among the wines.

ΔE^*_{ab}	Sweet RT	Sweet CR	Fortified RT	Fortified CR
Sweet RT		2.84	7.05	7.53
Sweet CR			9.21	9.60
Fortified RT				2.06
Fortified CR				

3. VOCs profile of Sweet and Fortified wines

Regarding VOC analysis (Table 6), a significantly lower amount of VOCs was measured in the Fortified wines across almost all compound classes: an 8–10 fold decrease for acids and a 20–50 fold decrease for alcohols, which are the two most abundant classes. The higher content in VOCs of Sweet wines is likely due to the fermentation process that is lacking in fortified wines. Only aldehydes were higher in Fortified wines, especially benzaldehyde, benzeneacetaldehyde, and syringaldehyde in CR wine. Benzaldehyde and benzeneacetaldehyde have a floral-fruity aroma (Kuang *et al.*, 2022) and are mainly produced during grape exposure to the sun (Liu *et al.*, 2024); their highest presence in Fortified wines, could be due to a greater extraction by ethanol from small debris in the must. It is known the affinity of ethanol for lignin (Hamzah *et al.*, 2021). The presence of syringaldehyde could be the result of lignin degradation due to alcoholic maceration, especially because the torque press

could have released a small portion of lignified and dried stem which was not blocked by the filter (Cernișev, 2017). Acetic acid was significantly reduced in Fortified wines and this was expected as aforementioned.

Comparing the wines from CR or RT dehydrated grapes, in Fortified wines, except for terpenes where the values were similar, the CR wines had higher concentrations in almost all other classes. In contrast, in Sweet wines, only the classes of alcohols and esters showed significantly higher content in the RT wines.

In Fortified wines, the CR sample had a higher content mainly due to acetic acid, phenylacetic acid, and palmitic acid whereas in Sweet wines the content of acids was similar in the two dehydration conditions. Even classes of alcohols and esters were in higher content in Fortified CR than in Fortified RT, meaning that the lower temperature allowed for preserving VOCs. However, these values were significantly lower than those from Sweet wines where the

TABLE 5. Single phenolic compounds (mg/L) in wines.

Phenolic compounds (mg/L)	Sweet RT	Sweet CR	Fortified RT	Fortified CR
Cyanidin 3-O-glucoside	2.96 ± 0.31 b	4.53 ± 0.35 a	<i>n.d.</i>	<i>n.d.</i>
Delphinidin 3-O-glucoside	8.53 ± 0.22 b	18.66 ± 0.79 a	0.75 ± 0.04 d	1.03 ± 0.14 c
Peonidin 3-O-glucoside	49.52 ± 1.73 a	8.28 ± 0.44 b	0.94 ± 0.04 c	<i>n.d.</i>
Malvidin 3-O-glucoside	45.38 ± 2.56 b	109.7 ± 9.71 a	3.01 ± 0.18 d	5.01 ± 0.79 c
Petunidin 3-O-glucoside	5.7 ± 0.87 b	12.91 ± 1.87 a	<i>n.d.</i>	<i>n.d.</i>
Total Anthocyanins	112.09 ± 1.14 b	154.08 ± 2.63 a	4.70 ± 0.19 d	6.04 ± 0.33 c
Myricetin	1.23 ± 0.21 a	0.34 ± 0.06 b	0.06 ± 0.02 c	0.03 ± 0.01 c
Quercetin	1.51 ± 0.12 b	4.15 ± 2.12 a	<i>n.d.</i>	<i>n.d.</i>
Quercetin 3-O-glucoside	7.73 ± 0.23 b	11.59 ± 1.02 a	0.47 ± 0.06 d	0.76 ± 0.09 c
Quercetin 3-O-rutinoside	1.19 ± 0.09 b	1.68 ± 0.04 a	0.05 ± 0.01 d	0.22 ± 0.04 c
Quercetin 3-O-ramnoside	0.61 ± 0.06 b	1.12 ± 0.09 a	0.24 ± 0.05 c	0.39 ± 0.12 c
Kaempferol	<i>n.d.</i>	0.22 ± 0.03 a	<i>n.d.</i>	<i>n.d.</i>
Total Flavonols	12.27 ± 0.12 b	19.10 ± 0.56 a	0.82 ± 0.04 d	1.40 ± 0.07 c
Catechin	25.34 ± 2.87 a	24.37 ± 1.01 a	5.67 ± 0.60 b	3.71 ± 0.18 c
(-)-Epicatechin	59.25 ± 3.24 b	99.55 ± 2.56 a	12.51 ± 0.95 d	29.55 ± 5.82 c
Procyanidin B1	5.33 ± 0.72 b	3.23 ± 0.21 c	0.07 ± 0.02 d	8.33 ± 0.34 a
Procyanidin B2	<i>n.d.</i>	<i>n.d.</i>	14.27 ± 0.21 b	21.96 ± 1.86 a
Procyanidin B3	12.34 ± 0.99 a	7.49 ± 0.07 b	0.32 ± 0.08 d	1.55 ± 0.11 c
Total Flavanols	102.26 ± 1.96 b	134.64 ± 1.06 a	32.84 ± 0.38 d	65.10 ± 1.66 c
<i>trans</i> Resveratrol-glucoside	0.33 ± 0.03 b	1.91 ± 0.09 a	<i>n.d.</i>	<i>n.d.</i>
<i>trans</i> Resveratrol	0.11 ± 0.03 b	0.62 ± 0.16 a	<i>n.d.</i>	0.16 ± 0.07 b
Total Stilbenes	0.44 ± 0.03 b	2.53 ± 0.16 a	<i>n.d.</i>	0.16 ± 0.07 c
Syringic acid	0.44 ± 0.10 b	0.83 ± 0.07 a	<i>n.d.</i>	<i>n.d.</i>
Gallic acid ethyl ester	3.14 ± 0.26 a	<i>n.d.</i>	<i>n.d.</i>	<i>n.d.</i>
Protocatechuic acid	<i>n.d.</i>	<i>n.d.</i>	1.09 ± 0.31 b	1.60 ± 0.14 a
Vanillic acid	2.41 ± 0.14 a	0.73 ± 0.08 b	<i>n.d.</i>	<i>n.d.</i>
4-Hydroxybenzoic acid	0.74 ± 0.06 b	9.93 ± 1.02 a	<i>n.d.</i>	<i>n.d.</i>
Gallic acid	2.06 ± 0.09 b	4.63 ± 0.03 a	0.94 ± 0.18 c	0.19 ± 0.03 d
Total Hydroxybenzoic acids	8.79 ± 0.12 b	16.12 ± 0.30 a	2.03 ± 0.20 c	1.79 ± 0.09 c

Data are the means (± standard deviation) of 3 wine reps. Different letters in each row indicate statistical differences ($p \leq 0.05$). *n.d.* = not detected.

RT sample had a greater concentration of alcohols and esters. The reason for a higher content of alcohols in Sweet RT is mainly due to phenethyl alcohol and 3-Methyl-1-butanol as a result of amino acid catabolism, which could be due to the higher presence of amino acids in dehydrated grapes at room temperature (optimal temperature for the metabolism) (Ossola *et al.*, 2017). Esters, and specifically ethyl palmitate, ethyl stearate, ethyl oleate, ethyl linoleate, and ethyl linolenate were higher in RT wines indicating a membrane lipid catabolism, which could suppose a greater fatty acid concentration, confirming what aforementioned for amino acids. Finally, terpenes were significantly higher in Sweet wines and this result could be due to the presence of solid must in fermentation, thus a greater extraction. In conclusion, regarding VOCs, grape dehydration at room temperature results in a more complex aroma due to fermentation, making this technique suitable for sweet wine production. On the

other hand, if fortification is the goal, dehydration at low temperatures is preferred.

4. E-nose analysis of Sweet and Fortified wines

PLS-DA stands as a robust multivariate analysis method widely employed in chemometrics to unravel relationships between input variables and categorical output variables. The score graph delineates the sample distribution within the space of latent variables (LVs), with each point signifying a sample's disposition relative to the LVs derived from PLS-DA. Here, 2 LVs were chosen to optimise the separation between different wine types, achieving 95 % explained variability (Figure 2).

Notably, the score plot vividly illustrates the separation between the Fortified and Sweet wine samples, transcending both CR and RT conditions. The clear segregation of these two groups

TABLE 6. Volatile organic compounds (VOCs) were detected ($\mu\text{g/L}$) in the wines. (part 1/3)

VOCs ($\mu\text{g/L}$)	Sweet RT	Sweet CR	Fortified RT	Fortified CR
Acetic acid	4643.94 $\pm$ 83.15 a	4456.19 $\pm$ 49.48 b	79.08 $\pm$ 1.69 d	190.01 $\pm$ 5.35 c
Isobutyric acid	112.93 $\pm$ 2.07 a	73.74 $\pm$ 0.27 b	11.36 $\pm$ 0.50 d	16.20 $\pm$ 0.79 c
Butanoic acid	26.99 $\pm$ 1.24 a	29.81 $\pm$ 6.55 a	7.10 $\pm$ 0.10 b	2.00 $\pm$ 0.20 c
Isovaleric acid	239.02 $\pm$ 1.75 a	211.99 $\pm$ 7.91 b	21.91 $\pm$ 0.22 d	61.65 $\pm$ 2.90 c
Pentanoic acid	<i>n.d.</i>	<i>n.d.</i>	16.99 $\pm$ 0.16 a	13.56 $\pm$ 0.30 b
Hexanoic acid	236.60 $\pm$ 2.66 b	412.66 $\pm$ 10.25 a	1.24 $\pm$ 0.17 c	0.77 $\pm$ 0.05 d
Heptanoic acid	<i>n.d.</i>	<i>n.d.</i>	17.19 $\pm$ 0.02 a	6.86 $\pm$ 0.57 b
<i>trans</i> -2-Hexenoic acid	4.93 $\pm$ 0.13 c	11.10 $\pm$ 1.80 b	18.55 $\pm$ 0.97 a	9.77 $\pm$ 1.62 b
Octanoic acid	728.44 $\pm$ 0.55 b	1207.21 $\pm$ 9.36 a	57.71 $\pm$ 4.30 c	63.06 $\pm$ 5.17 c
Nonanoic acid	9.75 $\pm$ 0.34 c	6.93 $\pm$ 0.29 d	51.05 $\pm$ 0.71 a	26.73 $\pm$ 1.35 b
Decanoic acid	351.44 $\pm$ 37.74 b	561.92 $\pm$ 1.78 a	68.91 $\pm$ 7.10 c	64.21 $\pm$ 0.79 c
Benzoic acid	54.07 $\pm$ 2.33 a	54.85 $\pm$ 0.28 a	22.42 $\pm$ 0.97 c	34.40 $\pm$ 6.27 b
Phenylacetic acid	198.60 $\pm$ 36.70 a	68.04 $\pm$ 24.22 c	33.95 $\pm$ 11.77 d	126.28 $\pm$ 7.10 b
Myristic acid	121.61 $\pm$ 40.57 a	87.26 $\pm$ 0.88 b	53.71 $\pm$ 0.24 d	63.40 $\pm$ 1.03 c
Pentadecanoic acid	65.49 $\pm$ 14.18 a	41.76 $\pm$ 2.16 b	36.58 $\pm$ 0.40 c	37.56 $\pm$ 0.69 c
Palmitic acid	818.27 $\pm$ 144.63 a	582.48 $\pm$ 63.73 b	293.30 $\pm$ 19.77 d	453.65 $\pm$ 48.94 c
Vanillic acid	204.03 $\pm$ 32.55 a	119.66 $\pm$ 32.01 b	<i>n.d.</i>	<i>n.d.</i>
Stearic acid	308.82 $\pm$ 77.68 a	188.58 $\pm$ 2.86 b	118.11 $\pm$ 1.44 c	127.30 $\pm$ 33.41 c
Total Acids	8124.94 $\pm$ 25.14 a	8114.18 $\pm$ 23.37 a	909.16 $\pm$ 12.97 c	1297.40 $\pm$ 10.74 b
Isobutyl alcohol	1226.92 $\pm$ 10.99 a	1013.45 $\pm$ 2.59 b	38.47 $\pm$ 3.52 c	39.02 $\pm$ 0.43 c
<i>n</i> -Butanol	59.48 $\pm$ 2.01 a	55.94 $\pm$ 0.81 b	6.96 $\pm$ 3.86 c	5.14 $\pm$ 1.56 c
3-Penten-2-ol	14.50 $\pm$ 0.09 a	14.50 $\pm$ 1.43 a	12.43 $\pm$ 0.04 c	12.89 $\pm$ 0.09 b
3-Methyl-1-butanol	14381.64 $\pm$ 37.70 a	13038.83 $\pm$ 10.05 b	169.12 $\pm$ 4.73 d	552.73 $\pm$ 14.37 c
3-Penten-1-ol	16.77 $\pm$ 1.60 a	12.37 $\pm$ 0.10 b	4.97 $\pm$ 0.15 c	5.07 $\pm$ 2.74 c
2-Hexanol	33.64 $\pm$ 1.16 bc	41.62 $\pm$ 3.09 a	29.92 $\pm$ 3.31 c	35.07 $\pm$ 2.83 ab
4-Methyl-1-pentanol	5.85 $\pm$ 0.17 a	5.85 $\pm$ 1.52 a	<i>n.d.</i>	<i>n.d.</i>
3-Methyl-1-pentanol	25.26 $\pm$ 0.12 a	21.86 $\pm$ 1.34 b	<i>n.d.</i>	<i>n.d.</i>
<i>n</i> -Hexanol	146.41 $\pm$ 0.13 b	297.82 $\pm$ 0.55 a	121.65 $\pm$ 0.62 c	87.18 $\pm$ 0.13 d
3-Ethoxy-1-propanol	11.15 $\pm$ 0.98 a	10.09 $\pm$ 1.27 a	<i>n.d.</i>	<i>n.d.</i>
3-Hexen-1-ol	14.63 $\pm$ 0.92 c	24.89 $\pm$ 0.45 b	38.94 $\pm$ 0.06 a	15.24 $\pm$ 0.44 c
2-Hexen-1-ol	<i>n.d.</i>	<i>n.d.</i>	103.25 $\pm$ 5.84 a	50.47 $\pm$ 0.37 b
2-Ethylhexanol	15.36 $\pm$ 1.78 c	19.49 $\pm$ 0.28 b	23.31 $\pm$ 2.12 a	16.74 $\pm$ 0.97 c
2-Nonanol	6.47 $\pm$ 0.80 ab	6.07 $\pm$ 0.52 b	5.56 $\pm$ 0.04 b	6.96 $\pm$ 0.21 a
Butane-2,3-diol	5140.67 $\pm$ 11.93 b	5488.17 $\pm$ 52.39 a	88.55 $\pm$ 2.75 d	187.88 $\pm$ 1.38 c
Propylene glycol	118.12 $\pm$ 4.72 b	140.73 $\pm$ 3.25 a	18.18 $\pm$ 1.19 d	21.95 $\pm$ 0.79 c
Methionol	42.90 $\pm$ 1.55 a	23.37 $\pm$ 4.68 b	<i>n.d.</i>	<i>n.d.</i>
2-Phenyl-2-propanol	<i>n.d.</i>	<i>n.d.</i>	1.20 $\pm$ 0.36 b	3.56 $\pm$ 0.28 a
Benzyl Alcohol	91.64 $\pm$ 2.45 a	59.30 $\pm$ 8.39 b	25.44 $\pm$ 0.79 d	29.36 $\pm$ 0.32 c
Phenethyl alcohol	34399.84 $\pm$ 586.41 a	17941.41 $\pm$ 796.89 b	388.07 $\pm$ 9.18 d	1740.96 $\pm$ 51.65 c
2-Phenoxyethanol	<i>n.d.</i>	<i>n.d.</i>	11.14 $\pm$ 2.56 a	11.44 $\pm$ 0.24 a
Tyrosol	3002.95 $\pm$ 722.71 a	1409.91 $\pm$ 99.20 b	<i>n.d.</i>	<i>n.d.</i>
Total Alcohols	58754.17 $\pm$ 169.78 a	39625.65 $\pm$ 164.72 b	1087.15 $\pm$ 92.30 d	2821.65 $\pm$ 74.47 c
<i>R</i> (+)-5,7-Dimethyl-1,6-octadiene	44.86 $\pm$ 3.43 b	62.11 $\pm$ 4.41 a	32.03 $\pm$ 0.28 c	19.64 $\pm$ 2.00 d
Linalool Oxide	21.99 $\pm$ 0.39 a	20.94 $\pm$ 1.39 a	7.88 $\pm$ 0.08 c	8.94 $\pm$ 0.36 b
<i>trans</i> <i>p</i> -Menthone	<i>n.d.</i>	<i>n.d.</i>	18.72 $\pm$ 0.31 a	18.24 $\pm$ 0.50 a
Linalool Oxide	14.04 $\pm$ 2.73 b	14.54 $\pm$ 1.43 b	14.22 $\pm$ 1.32 b	18.95 $\pm$ 0.14 a

TABLE 6. Volatile organic compounds (VOCs) were detected ($\mu\text{g/L}$) in the wines. (part 2/3)

<i>cis p</i> -Menthone	<i>n.d.</i>	<i>n.d.</i>	17.47 ± 0.69 a	17.64 ± 0.09 a
<i>L</i> -Linalool	208.71 ± 6.01 b	222.53 ± 10.56 a	79.52 ± 11.89 c	66.64 ± 2.17 d
<i>p</i> -Menth-3-ene	<i>n.d.</i>	<i>n.d.</i>	5.05 ± 0.69 b	7.04 ± 0.16 a
4-Terpineol	103.08 ± 6.53 c	153.10 ± 3.76 a	142.27 ± 5.23 b	74.04 ± 0.70 d
Ho-trienol	<i>n.d.</i>	<i>n.d.</i>	10.86 ± 0.05 b	11.09 ± 0.08 a
<i>p</i> -Menthan-1-ol	<i>n.d.</i>	<i>n.d.</i>	92.13 ± 2.25 a	98.97 ± 5.01 a
α -Terpineol	93.15 ± 1.14 a	96.83 ± 3.48 a	64.54 ± 1.53 b	57.32 ± 8.23 c
(+)- β -Citronellene	46.65 ± 0.52 a	43.74 ± 0.16 b	7.75 ± 0.18 c	8.59 ± 0.74 c
Epoxylinanol	<i>n.d.</i>	<i>n.d.</i>	32.66 ± 2.50 a	8.99 ± 0.12 b
β -Citronellol	90.95 ± 1.65 b	169.24 ± 12.40 a	7.02 ± 0.34 c	4.07 ± 0.03 d
<i>cis</i> -Geraniol	15.66 ± 0.71 c	21.83 ± 0.47 b	13.73 ± 3.23 c	36.99 ± 1.79 a
2,6-Dimethyl-3,7-octadiene-2,6-diol	158.67 ± 10.74 c	168.28 ± 12.83 b	171.96 ± 13.57 ab	171.85 ± 10.71 a
2,6-Dimethyl-7-Octene-2,6-diol	26.56 ± 4.50 a	25.89 ± 2.54 a	<i>n.d.</i>	<i>n.d.</i>
Terpene unknown	146.27 ± 32.02 a	60.60 ± 7.27 c	42.80 ± 2.09 d	90.32 ± 2.11 b
2,6-Dimethyl-1,7-octadiene-3,6-diol	100.74 ± 24.42 a	98.55 ± 9.29 a	57.13 ± 5.00 c	70.09 ± 3.08 b
Total Terpenes	1071.33 ± 48.68 a	1158.17 ± 50.46 a	817.74 ± 29.85 b	789.41 ± 22.11 b
Ethyl butyrate	58.13 ± 0.48 b	79.67 ± 0.70 a	<i>n.d.</i>	<i>n.d.</i>
Isoamyl acetate	238.81 ± 3.23 a	232.84 ± 2.50 a	<i>n.d.</i>	<i>n.d.</i>
Ethyl caproate	149.29 ± 0.71 b	289.24 ± 1.04 a	8.30 ± 0.34 c	7.49 ± 0.82 c
Ethyl lactate	153.36 ± 0.04 a	100.49 ± 0.86 b	3.51 ± 0.35 d	9.31 ± 1.11 c
Ethyl octanoate	290.64 ± 4.60 b	477.78 ± 0.15 a	8.09 ± 0.03 d	11.31 ± 0.39 c
Diethyl malonate	<i>n.d.</i>	<i>n.d.</i>	9.12 ± 0.79 b	12.47 ± 0.14 a
Ethyl 2-furoate	<i>n.d.</i>	<i>n.d.</i>	3.91 ± 0.11 a	2.98 ± 0.25 b
Decanoic acid, ethyl ester	632.66 ± 16.95 b	782.49 ± 24.78 a	10.97 ± 2.82 c	12.88 ± 1.62 c
Diethyl succinate	1358.64 ± 20.43 a	1113.39 ± 40.40 b	35.14 ± 0.68 c	33.92 ± 0.35 d
Ethyl 4-hydroxybutanoate	1308.84 ± 6.27 a	1083.67 ± 27.67 b	9.18 ± 0.01 c	6.52 ± 0.33 d
β -Phenylethyl acetate	134.39 ± 3.73 a	55.63 ± 2.33 b	<i>n.d.</i>	<i>n.d.</i>
Ethyl laurate	151.06 ± 6.03 b	181.09 ± 0.27 a	4.05 ± 0.47 d	5.67 ± 0.93 c
Diethyl DL-malate	221.01 ± 7.67 a	204.54 ± 3.25 b	181.84 ± 7.85 c	181.03 ± 5.36 c
Ethyl myristate	369.91 ± 22.55 a	279.44 ± 9.82 b	6.98 ± 2.10 d	32.81 ± 2.57 c
Ethyl cinnamate	<i>n.d.</i>	<i>n.d.</i>	19.44 ± 0.74 a	7.40 ± 1.85 b
Diethyl 2-hydroxypentanedioate	110.99 ± 4.51 a	71.66 ± 0.41 b	<i>n.d.</i>	<i>n.d.</i>
Ethyl palmitate	3758.13 ± 342.11 a	2748.34 ± 170.99 b	326.23 ± 7.65 d	549.56 ± 26.44 c
Ethyl hydrogen succinate	4195.65 ± 172.45 a	3706.33 ± 67.07 b	85.12 ± 11.56 d	247.07 ± 3.46 c
Ethyl stearate	1353.34 ± 150.37 a	809.60 ± 80.53 b	47.12 ± 3.47 d	104.91 ± 0.71 c
Ethyl oleate	1059.71 ± 241.44 a	552.34 ± 6.12 b	112.96 ± 0.88 d	166.85 ± 6.97 c
Ethyl linoleate	2153.10 ± 462.62 a	1604.07 ± 70.51 b	390.49 ± 3.75 d	504.78 ± 5.01 c
Ethyl linolenate	468.10 ± 87.11 a	270.03 ± 11.91 b	120.37 ± 0.04 c	118.87 ± 7.72 c
Methyl vanillate	58.18 ± 9.53 a	60.13 ± 0.19 a	9.51 ± 1.74 c	13.78 ± 1.21 b
Ethyl vanillate	122.24 ± 12.44 a	124.01 ± 2.81 a	55.83 ± 3.09 c	78.65 ± 0.41 b
Docosanoic acid, ethyl ester	261.81 ± 58.67 a	128.89 ± 7.19 b	<i>n.d.</i>	<i>n.d.</i>
5-Oxotetrahydrofuran-2-carboxylic acid, ethyl ester	138.20 ± 24.30 a	61.35 ± 12.48 b	<i>n.d.</i>	<i>n.d.</i>
Ethyl <i>p</i> -hydroxybenzoate	139.23 ± 2.44 c	99.86 ± 5.19 d	173.25 ± 6.96 b	195.40 ± 8.11 a
<i>p</i> -Hydroxycinnamic acid, ethyl ester	642.18 ± 2.91 c	1386.23 ± 162.51 a	259.72 ± 67.29 d	905.71 ± 94.74 b
Total Esters	19527.58 ± 65.74 a	16503.10 ± 56.47 b	1881.15 ± 19.44 d	3209.34 ± 44.11 c
Hexenal	<i>n.d.</i>	<i>n.d.</i>	22.66 ± 1.99 a	7.50 ± 0.23 b
Nonanal	6.86 ± 1.00 b	6.20 ± 0.71 b	7.23 ± 0.95 b	10.21 ± 0.13 a

TABLE 6. Volatile organic compounds (VOCs) were detected ($\mu\text{g/L}$) in the wines. (part 3/3)

Benzaldehyde	11.53 $\pm$ 0.45 c	8.72 $\pm$ 1.39 d	27.16 $\pm$ 0.74 b	118.69 $\pm$ 0.15 a
Benzeneacetaldehyde	<i>n.d.</i>	<i>n.d.</i>	16.45 $\pm$ 0.26 b	37.08 $\pm$ 3.81 a
Vanillin	102.13 $\pm$ 13.53 b	123.98 $\pm$ 10.57 b	81.58 $\pm$ 4.35 c	211.44 $\pm$ 3.90 a
Syringaldehyde	<i>n.d.</i>	<i>n.d.</i>	79.07 $\pm$ 14.39 b	167.41 $\pm$ 20.16 a
Total Aldehydes	120.52 $\pm$ 8.66 d	138.90 $\pm$ 7.56 c	234.15 $\pm$ 3.78 b	552.33 $\pm$ 14.73 a
3-Hydroxy-2-butanone	60.90 $\pm$ 3.06 c	109.94 $\pm$ 1.60 a	27.15 $\pm$ 1.54 d	71.39 $\pm$ 0.55 b
4-Hydroxy-4-methyl-2-pentanone	7.78 $\pm$ 0.13 c	9.38 $\pm$ 0.71 b	30.40 $\pm$ 1.80 a	3.55 $\pm$ 1.00 d
γ -Butyrolactone	130.17 $\pm$ 0.93 a	69.35 $\pm$ 8.48 b	8.11 $\pm$ 0.37 d	20.42 $\pm$ 0.28 c
Acetovanillone	82.18 $\pm$ 1.31 a	84.60 $\pm$ 3.98 a	6.48 $\pm$ 4.74 c	16.50 $\pm$ 1.12 b
Total Ketones	281.03 $\pm$ 1.36 a	273.27 $\pm$ 3.19 b	72.13 $\pm$ 2.11 d	111.86 $\pm$ 3.74 c
Undecane	74.07 $\pm$ 2.27 ab	76.10 $\pm$ 1.99 a	63.21 $\pm$ 0.42 c	72.40 $\pm$ 0.63 b
<i>cis</i> -5-Hydroxy-2-methyl-1,3-dioxane	44.75 $\pm$ 0.18 b	48.28 $\pm$ 0.30 a	<i>n.d.</i>	<i>n.d.</i>
<i>trans</i> -4-Hydroxymethyl-2-methyl-1,3-dioxolane	100.13 $\pm$ 0.55 a	102.83 $\pm$ 3.78 a	11.18 $\pm$ 0.53 c	8.70 $\pm$ 1.99 b
2,2-Diethoxyethyl-benzene	<i>n.d.</i>	<i>n.d.</i>	16.12 $\pm$ 0.42 b	46.60 $\pm$ 1.37 a
Damascenone	45.59 $\pm$ 1.16 b	42.21 $\pm$ 2.86 c	73.24 $\pm$ 3.48 a	43.93 $\pm$ 2.72 b
Benzothiazole	21.35 $\pm$ 1.51 a	20.80 $\pm$ 1.16 a	20.10 $\pm$ 1.97 a	21.14 $\pm$ 1.78 a
2,5-Dihydro-thiophene	121.88 $\pm$ 3.48 a	104.45 $\pm$ 0.37 b	62.77 $\pm$ 1.62 c	60.35 $\pm$ 1.11 c
2,3-Dihydro-benzofuran	73.09 $\pm$ 2.66 a	53.68 $\pm$ 2.37 b	40.50 $\pm$ 1.85 c	44.42 $\pm$ 2.24 c
3,4-Dihydro-8-hydroxy-3-methyl-1 <i>H</i> -2-benzopyran-1-one	162.97 $\pm$ 8.14 a	120.43 $\pm$ 3.46 c	98.29 $\pm$ 2.23 d	132.62 $\pm$ 7.33 b
5-Ethoxydihydro-2(3 <i>H</i>)-furanone	29.41 $\pm$ 0.19 a	23.31 $\pm$ 2.11 b	20.36 $\pm$ 2.08 b	14.46 $\pm$ 1.59 c
Total others	673.24 $\pm$ 4.35 a	592.07 $\pm$ 4.27 b	405.79 $\pm$ 7.47 d	444.63 $\pm$ 4.42 c

Data are the means ($\pm$ standard deviation) of 3 wine reps. Different letters in each row indicate statistical differences ($p \leq 0.05$). *n.d.* = not detected.

into opposing quadrants underscores significant aromatic differences between the samples. Sweet wines were also discriminated against for the two dehydration methods while Fortified wines were not, suggesting a high similarity between the two Fortified wines. The variable important in projection (VIP) gives information on the sensors responsible for the segregation of the scores (Figure 3). Notably, sensor 7 emerges as a key discriminator, particularly influential in delineating Sweet RT wine from its counterparts. Conversely, sensors 2, 3, 4, and 6 play a more prominent role in distinguishing Fortified wine. Particularly noteworthy is the association between sensor 4 (QMB4) and certain aromatic compounds, including volatile phenols and furan compounds (Martínez-García *et al.*, 2021; Saevels *et al.*, 2004), as corroborated by prior studies employing a similar QMB-E-nose configuration. Similarly, sensor 7 has been linked to aldehydes and alcohols in existing literature (Martínez-García *et al.*, 2021; Saevels *et al.*, 2004), suggesting that these classes have particular significance in discriminating between wine types. Aldehydes and alcohols were indeed different in the different wines with Fortified wines having lower content of some alcohols (such as 3-methyl-1-butanol and phenylethanol) and Sweet RT wines containing higher concentrations of 3-methyl-1-butanol and phenethyl alcohol.

The statistical robustness of the PLS-DA model is also evaluated through a confusion matrix, along with related

efficiency and robustness indices in calibration and cross-validation (Table S1). During calibration, the model achieves 100 % accuracy in identifying the four classes. In the cross-validation phase, the model maintains good statistical efficiency in discriminating the sweet wines (with a maximum error of 0.08). On the other hand, misclassifications were observed for Fortified wines with CT samples assigned to the Fortified RT class, reinforcing the similarities already observed in the score plot. Even though these two wines presented different analytical aromatic profiles, the slight discrepancies between GC-MS and E-nose measurements are not completely unexpected. Hence, GC-MS gives the precise quantification of specific VOCs present in the analysed matrix but it does not take into account chemical interactions, combinations, and synergy between different molecules (Tari *et al.*, 2024). Each sensor in the E-nose array responds differentially to different VOCs, creating a characteristic fingerprint of the sample's volatile composition. The combined sensor responses generate a dataset representing the overall VOC profile (Modesti *et al.*, 2024).

QMB-based e-nose has been already used to discriminate wines from different varieties and vintages (Alfieri *et al.*, 2024; Harris *et al.*, 2023), to identify off-flavours formation (Cynkar *et al.*, 2007), and to differentiate different sweet wines (García-Martínez *et al.*, 2011), highlighting its potential.

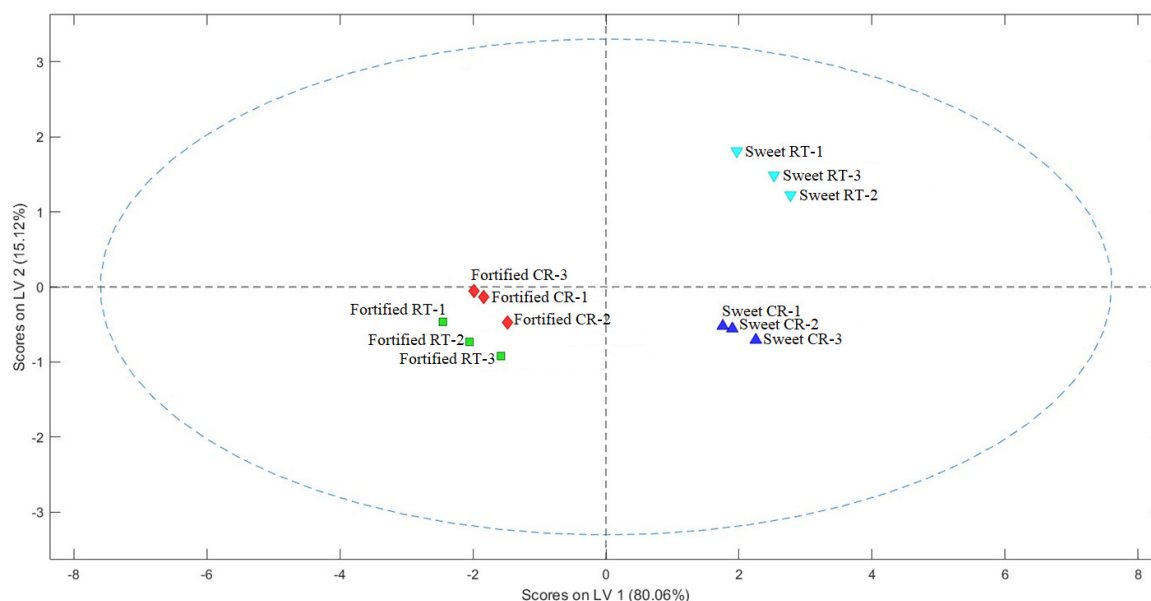


FIGURE 2. Score plot (LV1 vs LV2) of the Partial Least Squares Discriminant Analysis (PLS-DA) model built from the data obtained from the measurements with the electronic nose (the numbers 1, 2, 3, indicate the three bottles used for the analyses).

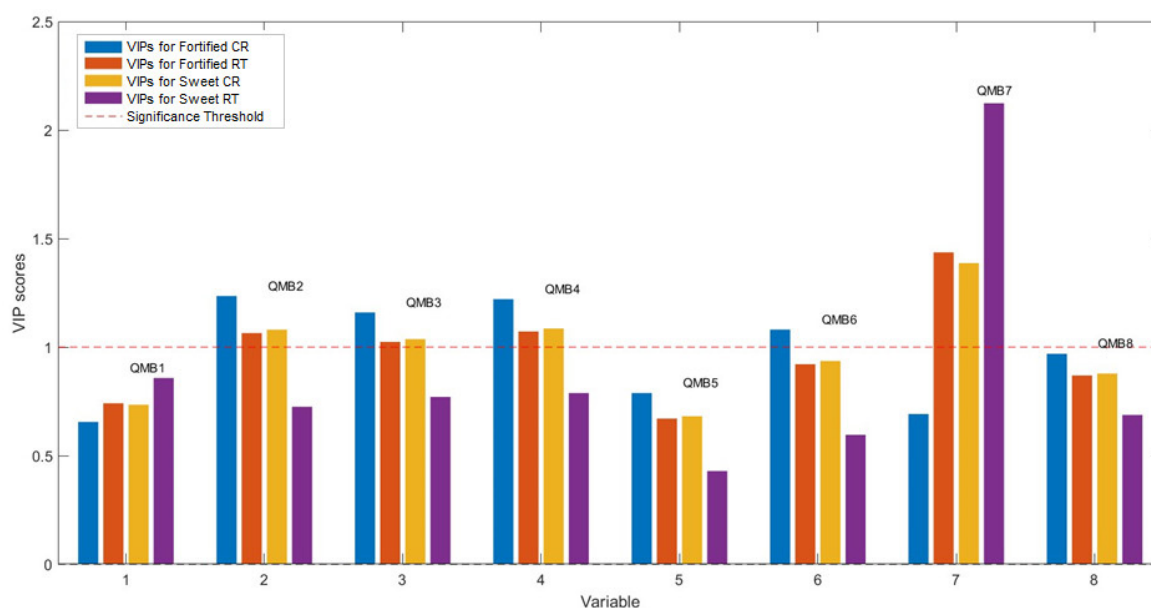


FIGURE 3. Effect of variables (loadings, QMB sensors of the Nose) on the pattern recognition discrimination of the PLS-DA operated on the acquisitions of the headspaces at the electronic nose. Influences are evaluated as a projection of the VIPs (variable importance projections) generated by each variable (QMBs) and evaluated based on an efficacy threshold (equal to 1).

However, other studies suggest that the E-nose systems still present important limitations, especially low selectivity, which make it hard to discriminate different samples when the aromatic fingerprints are not so different (Celdrán *et al.*, 2022). In this context, it can be hypothesised that the addition of ethanol in fortified wines complicates the E-nose's ability to distinguish between the two wines, as ethanol itself may mask other differences caused by varying dehydration rates. Hence, ethanol generates a strong signal on sensor arrays, which can impair the ability of E-noses to discriminate between different

aromas. Moreover, the presence of ethanol can decrease the volatility of other aroma compounds, further complicating the discrimination process (Ragazzo-Sanchez *et al.*, 2006).

5. NIR-AOTF analysis of Sweet and Fortified wines

The PLS-DA obtained with the transformed spectra (absorbance = $\log 1/T$), described 97 % of the total variability across the first two latent variables (LV1 82.15 % and LV2 15.25 %) (Figure 4).

In the score plot, the Sweet wines cluster together regardless of the CR or RT conditions, suggesting a high similarity between them. On the other hand, Fortified wines exhibit clear separation from each other, occupying opposing quadrants. In NIR spectroscopy, spectral absorbances are directly linked to fundamental molecular vibrations (overtones and combination bands) of organic functional groups such as C-H, N-H, O-H, and S-H (Rouxinol *et al.*, 2022). The vibrations of chemical bonds in complex matrices occur at specific frequencies, determined by the mass of the atoms, the stiffness of the bonds, and vibrational coupling effects. As a result, most biochemical and chemical species in wine exhibit characteristic absorption bands in the NIR spectral regions. Therefore, the analysis of absorbance peaks in the VIP plot (Figure 5) allows for the identification of significant spectral ranges responsible for the discrimination between different wines, enabling some important considerations of the organic molecules involved (Cozzolino, 2015).

For instance, in the Fortified CT wine, the first significant absorption bands have been observed in the spectral region around 1200 nm as well as around 1400 nm, which can be attributed to sugar-related absorption bands (González-Caballero *et al.*, 2010). Moreover, the higher absorption observed in the spectral portion related to 1400–1450 nm, can be associated with water molecules (Cozzolino *et al.*, 2006), due to the first overtone of O-H stretch and the O-H asymmetric stretching and bending combination (Osborne *et al.*, 1993). The higher absorption in the spectral region around 1200–1400 nm is especially relevant, as fortified wines consistently show elevated concentrations of alcohol, from the addition of ethanol, and higher sugars, due to the lack of fermentation. Thus underscoring the capability of NIR spectroscopy to

detect the vibrational modes of these molecules. A minor spectral modification can be also observed between the 2000–2200–2400 nm portion, potentially assignable to phenol and condensed tannin (Cozzolino *et al.*, 2004; Dykes *et al.*, 2014; Zhang *et al.*, 2008). This spectral feature well correlates with the differences observed in anthocyanins and polyphenols observed in the wines (significantly lower in Fortified wines compared to Sweet wines).

Notably, the constructed model demonstrates good performance in class assignments, achieving 100 % accuracy and error equal to 0 in both calibration and cross-validation phases (Table S2). The robustness of the model underscores the reliability of NIR in accurately discriminating the diverse classes. The excellent assignment pattern and the observed absorption bands effectively correspond to compositional behaviours, such as those related to sugars and polyphenols. However, the NIR approach, while effective in class discrimination, can be complex due to the overlapping interactions between absorption bands and the intrinsic characteristics of the wine matrix, making it challenging to establish a direct and precise link to the concentrations of specific compounds (Littarru *et al.*, 2025).

CONCLUSIONS

Fortification is a common technique to produce highly prized sweet wines subjected to long ageing. The addition of alcohol can be also a shortcut to producing low-priced sweet wines, which in Italy are called liquorous. In this paper, two techniques of grape dehydration used to produce sweet wines with or without alcohol addition were compared. Cold room dehydration provided better quality grapes with

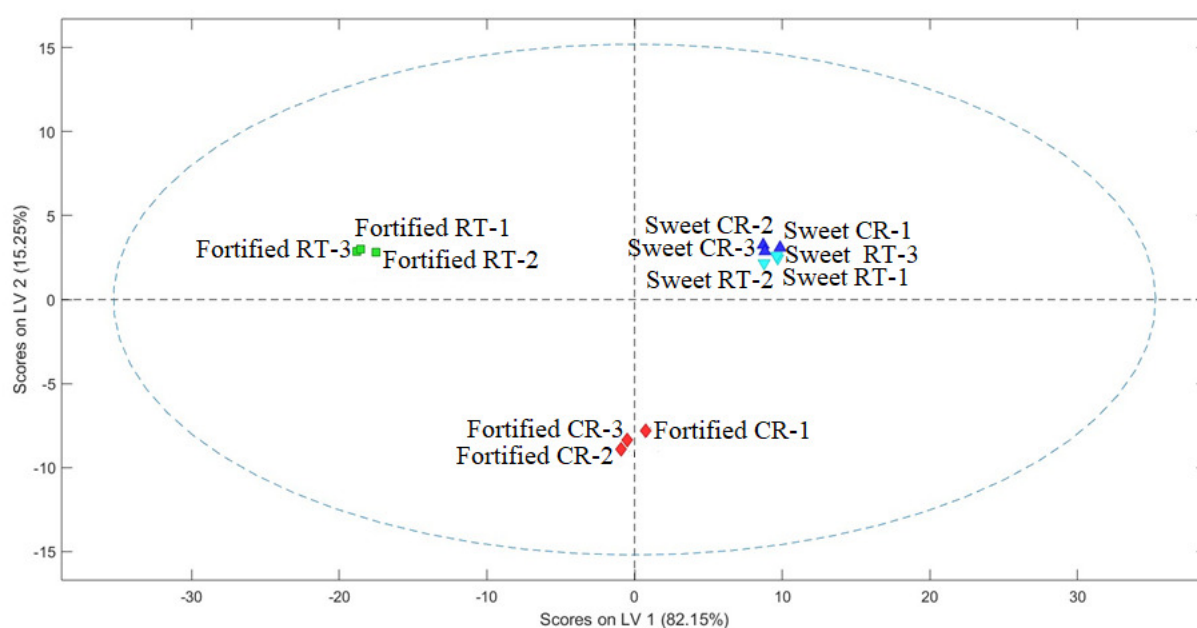


FIGURE 4. Score plot (LV1 vs LV2) of the Partial Least Squares Discriminant Analysis (PLS-DA) model constructed from data obtained from NIR-AOTF spectral acquisitions (the numbers 1, 2, 3, indicate the three bottles used for the analyses).

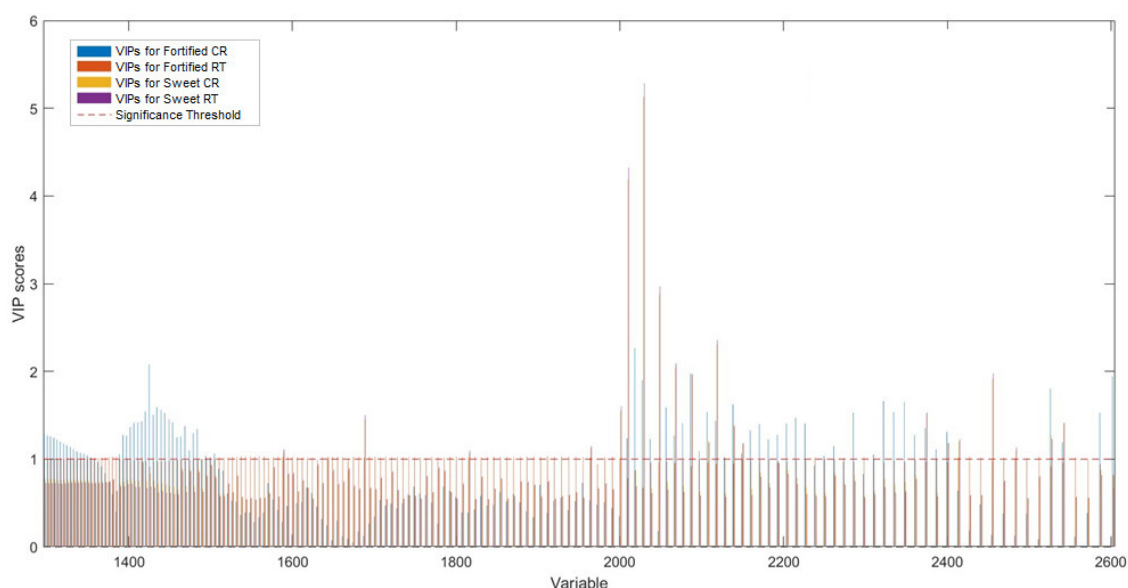


FIGURE 5. Effect of variables (loadings, wavelengths) on the pattern recognition discrimination of the PLS-DA operated on spectral acquisitions of NIR-AOTF. Influences are evaluated as a projection of the VIPs (variable importance projections) generated by each variable (wavelengths) and evaluated based on an efficacy threshold (equal to 1).

higher concentrations of all classes of analysed phenols. Fortification produced wines with lower glycerol and volatile acidity whatever the dehydration technique was but with a much lower content in polyphenols and overall in VOCs.

The E-nose approach was able to correctly discriminate between the Sweet and Fortified wines. For the sweet wines the correct assignment was also observed between control and cold room dehydration suggesting important aromatic differences between the two wines. However, in the case of fortified wine important misclassification was observed. The alignment between E-nose measurements and GC-MS analyses was not always straightforward, likely due to the different nature of these techniques. While GC-MS quantifies specific VOCs, the E-nose gives an overall volatile fingerprint. Furthermore, the high ethanol content in fortified wines may interfere with the E-nose's ability to accurately capture volatile profiles, complicating the interpretation of aromatic characteristics. This discrepancy suggests that although the E-nose can be a valuable tool for sample discrimination, it does not always provide direct compositional information, especially in matrices with high ethanol concentrations. On the other hand, NIR-AOTF correctly assigned the different wines to their respective classes indicating high precision of vibrational spectroscopy, probably linked to changes in wine matrix composition such as differences in sugar, alcohol, and polyphenol content, accordingly with the absorbance bands. However, despite the high classification accuracy of the model, the direct correlation between spectral features and specific compositional differences remains challenging. Overall, both E-nose and NIR-AOTF offer pattern recognition rather than precise compositional analysis, requiring cautious interpretation. Further studies are needed to refine their application and strengthen correlations with chemical

composition. Nonetheless, these tools hold promise for the industry, enabling faster and more efficient wine monitoring.

In conclusion, this study provides valuable insights into the impact of different dehydration techniques on the production of both sweet and fortified wines, offering guidance for winemakers in selecting the most suitable approach based on their desired final product. Cold room dehydration proved to be the most effective method for enhancing grape quality, leading to a higher concentration of phenolic compounds and volatile organic compounds, which are crucial for the sensory profile of sweet wines. Conversely, fortification, regardless of the dehydration method, resulted in wines with improved stability and lower volatile acidity, making it a viable strategy for the production of long-aged wines. These findings highlight how dehydration and fortification choices should be tailored to the specific characteristics and market positioning of the final product, optimising both quality and production efficiency.

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