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How does the application of ultrasound energy influence the ageing of a bottled red wine?

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ABSTRACT

A red wine that had aged in bottle for five years was treated with ultrasound (40 kHz) for 5 min and 30 min twice weekly and analysed after 3, 6, 9, and 12 months. Sensory analysis showed differences in overall quality at months 3 and 6, with the ultrasound-treated wines preferred. The ultrasound treatment did not decrease the free sulfur dioxide content. Differences in anthocyanins, polyphenols, and proanthocyanidins were clearest at month 9. In contrast, differences in the volatile profile were clearest at month 3. From a commercial point of view, the low-energy treatment (5 min) might be preferable given the lower costs. Ultrasound treatment was effective in enhancing the overall quality during the first 6 months of bottle storage in an aged red wine and did not increase the risk of oxidation or microbial spoilage. This work made it possible to fine-tune ultrasound treatment to maximise its positive effects.

1. Introduction

Fine wines are admired for their ability to age. It has long been known that ageing fine wine under controlled conditions can increase both the complexity of its sensory characteristics and its potential commercial value (Bettinson et al., 2019). Unfortunately, the process of ageing wine is slow and thus expensive. This makes the process uneconomic for wineries at most price segments. Therefore, there is a potential demand for technologies that could increase the rate of wine ageing for commercial purposes.

As wine ages, it undergoes characteristic changes in chemical and sensory properties. A distinction should be made between oxidative ageing (maturation), generally carried out in a barrel (or with micro-oxygenation devices for red wines) and reductive ageing (García Martín & Sun, 2013). Typical changes for red wines during the ageing process include a change in colour from purple to ruby, then garnet up to brick red, a softer, gentler flavour profile, and the emergence of tertiary flavours and aromas. These changes are typically due to the polymerisation of anthocyanins and colourless flavonoids. Consequently, the presence of derived pigments such as vitisins is considered characteristic of aged wines (Alcalde-Eon et al., 2006; Bettinson et al., 2019; Chira et al., 2011). Other changes in aroma chemistry that are typical for aged wines can include the formation of ethyl esters of fatty acids and higher alcohol acetates (Antalick et al., 2014; Bettinson et al., 2019). Aged wines are often more complex in flavour and aroma profile, and thus are considered more desirable by educated consumers (Bettinson et al., 2019).

The desire to mature or age wine more rapidly is not new. Bettinson et al. (2019) cited Galen (129 CE-c. 216 CE) as stating that 'aged' wine need not be old but can be a wine that has the characteristics associated with aged wines. In the last decades, several new and/or alternative techniques have been investigated to age wine more rapidly. One of the most well-known methods is micro-oxygenation, an oxidative maturation method that has gained widespread commercial acceptance (Gómez-Plaza & Bautista-Ortín, 2019; Solar et al., 2023). The use of oak staves, chips, or shavings as a rapid and inexpensive method to simulate barrel maturation is also widespread (Dambergs et al., 2012; Gómez-Plaza & Bautista-Ortín, 2019; Solar et al., 2023), but is generally considered to be lower in quality.

To mimic reductive ageing, the use of methods such as high pressure, pulsed electrical fields, microwaves, gamma radiation, and ultrasound have all been trialled (García Martín & Sun, 2013; Solar et al., 2023). Currently, commercial use is limited due to legislation and consumer resistance. In addition, only certain wine styles will age well, and thus

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only certain wine styles would benefit from rapid ageing with ultrasound treatment. Generally, wines that are low in pH and high in flavour compounds and phenolics such as tannins are considered to have the most ageing potential (Bettinson et al., 2019).

Ultrasound refers to sound waves with a frequency of 20 kHz or above that cannot be detected by humans (Gavahian et al., 2022; Solar et al., 2023). Previous studies have examined its use in processing red grape musts (Celotti et al., 2023; Ferraretto et al., 2013; Martínez Lapuente et al., 2021; Natrella et al., 2023; Pérez-Porras et al., 2022; Sánchez-Córdoba et al., 2021), white grape musts (Celotti et al., 2023; Gómez-Plaza et al., 2022; Labrador Fernández et al., 2022), wine (Ahmad et al., 2023; Ferraretto & Celotti, 2016; Focea et al., 2023; García Martín & Sun, 2013; Luchian et al., 2024; Sánchez-Córdoba et al., 2021; Singleton & Draper, 1963; Tao et al., 2014b, 2014c), and wine-based products such as brandy and vinegar (Sánchez-Córdoba et al., 2021).

It is suggested that wine aged rapidly through the application of ultrasound can be of excellent quality (Ferraretto & Celotti, 2016; Solar et al., 2023; Tao et al., 2014a, 2014b). The OIV has authorised the use of ultrasound for processing crushed grapes (must) in Resolution OENO 616–2019, but not for processing finished wine (OIV, 2019). Martínez Lapuente et al. (2021) noted an increased impact on wine monosaccharides and grape polysaccharides at 28 kHz, as compared with 20 kHz. When utilised during winemaking, the effect likely differs between cultivars (Natrella et al., 2023).

There is less published work on the impact of ultrasound on red wine ageing than on the impact on vinification, but it is hypothesised that frequencies between 20 and 100 KHz could accelerate the ageing process (García Martín & Sun, 2013). It is thought that acoustic cavitation caused by sonication may cause the formation, growth, and violent collapse of voids or small bubbles. The collapse of these bubbles can produce localised very high temperatures and pressures, and the consequent production of free radicals. These conditions can cause polymers to break up and recombine, enhancing the flavour and body of the wine (Gavahian et al., 2022; Singleton & Draper, 1963; Solar et al., 2023; Zhang et al., 2023; Zheng et al., 2014). It may also accelerate the oxidation, polymerisation, and condensation of aldehydes, alcohol, olefins, and esters (Zheng et al., 2014).

Consequently, ultrasound may be able to reduce the astringency of red wine without impacting wine colour (Ahmad et al., 2023; Natolino & Celotti, 2022), improving consumer acceptance. Singleton and Draper (1963) noted, however, that ultrasound-induced reactions are generally not easily reproducible. The use of ultrasound in ageing wines was discussed in review papers by García Martín and Sun (2013), Gavahian et al. (2022), and Zhang et al. (2023).

It is possible, therefore, that ultrasound could be suggested for use as an innovative, technical method to accelerate the ageing process of wine. If so, this would allow for wineries to release wine that has been optimally aged without the expense and time associated with traditional bottle ageing methods (García Martín & Sun, 2013). This is particularly of interest given that few consumers store wine for more than two weeks prior to consumption (Thach & Camillo, 2018), meaning that it is not practical for most wineries in most markets and price segments to rely on consumer willingness to age wines. This technology would thus provide an innovative and cost-effective method for wineries to enhance the ageing of bottled wines before commercialisation.

This study is distinct from previous studies in its use of a wine that had already been aged for five years. Most previous studies reporting ultrasound treatment of finished wines have examined its impact on young wines. Given that a wine of this age would already be expected to have reached some degree of chemical equilibrium, the impact of the ultrasound treatment is not obvious. Additionally, the use of liquid chromatography-mass spectrometry (LC-MS), bidimensional gas chromatography-mass spectrometry (GC×GC–MS), and sensory analysis in combination is novel to this study and allows for a more thorough and robust analysis of the data.

Regular application of ultrasound at a fixed duration and intensity to bottled red wines was applied over 12 months and both sensory and chemical analysis of the wine was conducted every three months. The study aimed to evaluate the changes in the chemical and sensory profile and panel overall quality rating resulting from ultrasound treatment.

2. Materials and methods

2.1. Wine samples and ultrasound treatment

Ultrasound treatment was conducted twice weekly throughout the entire study period using a Vevor Professional Ultrasonic Cleaner JM100 with 30 L capacity and 6oo W ultrasonic power at 40 kHz (Rancho Cucamonga, CA, USA). Both low-energy (5 min treatment time) and high-energy (30 min treatment time) treatments were conducted, allowing for fine-tuning of the operating parameters required for ultrasonic treatment. The water temperature of the bath was kept below 23 °C during treatment via cooling using a MPM M408-BC cryostatic bath (MPM Instruments SRL, Bernareggio, MB, Italy).

The wines were analysed via a sensory panel and analytical instrumental techniques four times throughout the year. These were T03: 3 months of ultrasound treatment (5th July 2022), T06: 6 months of ultrasound treatment (5th October 2022), T09: 9 months of ultrasound treatment (5th January 2023), and T12: 12 months of ultrasound treatment (5th April 2023). The sampling plan is shown in Table 1.

The wine used for the study was a 2017 Club del Buttafuoco Storico 'I Vignaioli del Buttafuoco Storico' (DOC 'Buttafuoco Storico') provided by Consorzio Club del Buttafuoco Storico (Canneto Pavese, PV, Italy). This was a bottled wine with 15 % alcohol. The blend was 50 % Croatina and 25 % Barbera, with the remaining 25 % Ughetta di Canneto and Uva Rara.

2.2. Sensory analysis

Sensory analysis was undertaken at T03 using the triangle test and at T06, T09, and T12 using Quantitative Descriptive Analysis (QDA®) (Stone et al., 1974). 'Sensy', a web-based application developed in collaboration with the Computer Science Faculty of the Free University of Bozen-Bolzano as part of the WineID project (TN201-ID2019), was used to gather responses from panellists during both training and data collecting sessions.

At T03 a triangle test was performed to provide a rapid assessment regarding whether differences between samples were noticeable to consumers. 18 consumers aged from 25 to 55 years old, of whom 70 % were female and 30 % male. Each consumer was shown each sample twice. For an α risk of 0.05 with 18 consumers, the minimum number of consumers who could detect the odd sample is 10 (Rogers, 2017).

At T06, T09, and T12, QDA was undertaken. QDA was conducted as described in Dupas de Matos et al. (2020). The QDA panellists ranged in age from 27 to 46 years of age, of whom 66 % were female and 33 % male. The sensory attributes used for QDA were determined by panellists at a roundtable session, based on the work of Noble et al. (1987). The panel was subsequently trained on the selected sensory attributes, with a total of ten training sessions involving the use of commercially obtained aroma standards ('Nez du Vin', Éditions Jean Lenoir, Cassis, France) and

Table 1
Treatment numbers and times. Low energy: 5 min treatment time, high energy: 30 min treatment time.

	T0 (05/04/2022)	T03 (05/07/2022)	T06 (05/10/2022)	T09 (05/01/2023)	T12 (05/04/2023)
Control	3	3	3	3	3
Low Energy	N/A	3	3	3	3
High Energy	N/A	3	3	3	3

wine samples prepared with samples of relevant natural food products. In addition, panellists were asked to give an overall quality judgement that took into account all of the sensory attributes measured and was a judgement of the intrinsic quality of the wine rather than personal liking. A table of sensory attributes can be found in Supplementary Table S1 in the supporting information.

Subsequently, the wines were assessed at the relevant times, and sensory attributes were rated on a scale from zero to nine. The questionnaires used for the analysis were delivered online via Sensy, a web-based application. Panellist performance was assessed before statistical analyses, and outliers (panellists whose scores were outside ±2 of the mean for five or more sensory attributes, based on a Tukey test) were discarded from the results.

Participants gave informed consent, and an affirmative reply was required to enter the survey. They were able to withdraw from the survey at any time without giving a reason and without any disadvantage to themselves. The privacy rights of all subjects were observed. The panellists were all regular wine consumers recruited voluntarily from the Free University of Bozen-Bolzano and NOI Techpark (Bolzano, Italy), and were not compensated. For QDA, panellists were given a maximum of nine 30-mL wine samples plus one 30 mL calibration sample per sensory session (equivalent to 39.3 mL of pure ethanol) and were encouraged to taste the sample without swallowing. The products tested were safe for consumption, the study was conducted following the Declaration of Helsinki (World Medical Association, 2013), and all procedures were performed in compliance with relevant laws and institutional guidelines.

2.3. Wine colour (CIELab) analysis

CIELab is a three-dimensional representation that aims to capture the multidimensional gamut of human colour perception (Durmus, 2020; Sant'Anna et al., 2013). It defines a three-dimensional colour space where L^* represents lightness (L^* 0 = black, L^* 100 = diffuse white), a^* represents green to red (negative values indicate green, positive values indicate red), and b^* represents blue to yellow (negative values indicate blue, positive values indicate yellow) (Bakker et al., 1986; Duley, 2021; Liang et al., 2011). C^* (chroma) and H (hue) are derived from a^* and b^*, and can be calculated as described by Bakker, Bridle and Timberlake (1986). Colour was analysed using a Konica Minolta CR-400 (Chiyoda City, Tokyo, Japan) colorimeter for CIELab colour space.

2.4. Wine chemical analysis

Wine quality was evaluated for standard oenological parameters using a multiparametric wine analyser (MIURA One, Exacta+Optech, San Prospero, Italy). Measurements were taken of tartaric acid, malic acid, acetic acid, free sulfur dioxide, and total sulfur dioxide levels.

Analyses of anthocyanins, phenols, and oligomeric condensed tannins (proanthocyanidins, PAC) were conducted using an Agilent LC-QqQ 6465 system (Agilent, Santa Clara, CA, USA) equipped with a 1260 Infinity II UHPLC with a quaternary pumps system, a 1260 Infinity II WR PDA detector, in series to a AJS (ESI) QqQ-MS mass analyser. Compounds were tentatively identified based on fragmentation studies and on comparison with the literature.

Anthocyanins were measured as per Darnal et al. (2023b). Briefly, separation was conducted using a Poroshell 120 (Agilent Technologies, Santa Clara, CA, USA) at 30 °C, with a flow rate of 0.35 mL min^{-1}. The mobile phases were phase A (4.5 % formic acid in ultrapure water) and B (4.5 % formic acid in acetonitrile), with a gradient separation programme of 5 % B from 0 to 1 min, 5 to 15 % B from 1 to 10 min, 15 to 25 % B from 10 to 15 min, 25 to 40 % B from 15 to 18 min, 40 to 95 % B from 18 to 21 min, 95 % B from 21 to 24 min, 95 to 5 % B from 24 to 25 min, 5 % B from 25 to 28 min. The photodiode array detector recorded absorbances from 200 to 700 nm with a 4 s response time, a 4 nm slit width, and 1 nm spectrum steps. The MS detector was set to ESI+ ionisation mode, with the following parameters: capillary voltage, + 3500 V; cell acceleration, 5 V; fragmentor potential, 135 V; mass range, m/z 200–700; N_2 gas temperature, 340 °C; N_2 gas flow, 13 L. min^{-1}; nebuliser pressure, 50 psi; nozzle voltage, + 1000 V; scan time, 500 ms; sheath gas flow, 12 L.min^{-1}; sheath gas heater, 350 °C; step size, 0.1 amu.

PAC were measured as described by Darnal et al. (2023a). Briefly, separation was carried out using a Vertex Plus Eurospher II (Knauer, Berlin, Germany) column and precolumn at 30 °C, with a flow rate of 0.7 mL min^{-1} and an injection volume of 5 μL. The mobile phases were phase A (0.1 % formic acid in ultrapure water) and B (0.1 % formic acid in acetonitrile), with a gradient separation programme of 1 % B from 0 to 2.5 min, 1 to 25 % B from 2.5 to 50 min, 25 to 99 % B from 50 to 51 min, 99 % B from 51 to 55 min, 99 to 1 % B from 55 to 56 min, and 1 %B from 56 to 62 min. The MS was operated in ESI+ ionisation mode, with single ion monitoring mode, with the following parameters: capillary voltage, + 3000 V; cell acceleration, 5 V; fragmentor potential, 135 V; N_2 gas flow, 8 L min−1; N_2 gas temperature, 230 °C; nebuliser pressure, 20 psi; nozzle voltage, 2000 V; sheath gas flow, 10 L min−1; sheath gas heater, 300 °C.

Polyphenols were measured as per Poggesi et al. (2022). For separation, a Poroshell 120 column (Agilent Technologies) was used at 30 °C, with a flow rate of 0.35 mL min^{-1} and an injection volume of 5 μL. The mobile phases were phase A (0.1 % formic acid in ultrapure water) and phase B (0.1 % formic acid in acetonitrile), and a gradient separation programme of 1 % B from 0 to 1.5 min, 1 to 30 % B from 1.5 to 19 min, 30 to 99 % B from 19 to 20 min, 99 % B from 20 to 24 min, 99 to 1 % B from 24 to 25 min, and 1 % B from 25 to 30 min. The photodiode array detector recorded absorbances from 200 to 700 nm with a 4 s response time, a 4 nm slit width, and 1 nm spectrum steps. The MS detector was set to ESI- ionisation mode, with the following parameters: capillary voltage, - 3500 V; cell acceleration, 5 V; fragmentor potential, 135 V; mass range, m/z 200–750; N_2 gas temperature, 340 °C; N_2 gas flow, 13 L.min^{-1}; nebuliser pressure, 50 psi; nozzle voltage, −500 V; scan time, 500 ms; sheath gas flow, 12 L min^{-1}; sheath gas heater, 350 °C; step size, 0.1 amu.

Analysis of volatile compounds was conducted using a GC×GC coupled with a Pegasus BT 4D time-of-flight mass spectrometer and equipped with a Flux™ flow modulator (Leco, St. Joseph, MI, USA). GC×GC–MS was as per Poggesi et al. (2022). Briefly, samples were prepared by adding 0.5 g of NaCl and 10 μL of 2-methyl-3-pentanol (internal standard, from a stock of 1/50 of IS in ethanol) to 4 mL of wine. Solid phase microextraction was performed using a Stableflex triphasic SPME fibre (SUPELCO, Bellefonte, PA, USA) for the autosampler and a LPAL3 GC autosampler (LECO) equipped with a Peltier Stack, with 5 mins incubation and 60 min extraction time at 50 °C and 300 rpm. The fibre was preconditioned for 6 mins at 240 °C. The separation was carried at 1 mL·min^{-1} flow rate at constant flow mode. Separation was performed using a polar cross-bond PEG-phase MEGA-Wax Spirit (Mega S.r.l., Legnano, MI, Italy) as the first dimension and a Rxi-17 Sil (Restek Corporation, Bellefonte, PA, USA) as the second dimension connected by a Flux modulator (LECO). The temperature programme for the primary oven was 40 °C for 6 min, 40 to 180 °C at 3 °C min^{-1}, 180 to 240 °C at 10 °C min^{-1}, and 240 °C for 1 min, and the secondary oven was set at 5 °C higher than the primary oven. The GC×GC and MS were connected with a transfer line set to 250 °C. The MS detector was set with the following parameters: acquisition delay, 20 s; acquisition mass range, 35–530 m/z; extraction frequency, 32 kHz; filament electron energy, 70 eV; ion source temperature, 250 °C; and spectra s^{-1} acquisition rate, 150. Peaks were aligned using ChromaTOF software (Leco), and compound assignment was carried out automatically via database comparison using NIST 2017 (Darnal et al., 2023c).

2.5. Statistical analysis

Principal Component Analysis (PCA) was conducted using GNU R

4.3.3 (R Core Team, 2024) with the 'PCA' functions of the 'FactoMineR' (Lê et al., 2008) and 'factoextra' packages (Kassambara & Mundt, 2020) or the 'dudi.pca' function of the 'ade4' package (Bougeard & Dray, 2018; Thioulouse et al., 2018) under Microsoft Windows 10. Multiple Factor analysis (MFA) was conducted using GNU R 4.3.1 with the 'MFA' functions of the 'FactoMineR' (Lê et al., 2008) and 'factoextra' packages under Microsoft Windows 10.

The triangle test analysis was conducted using GNU R 4.3.1 with the 'triangle.pair.test' function of the 'SensoMineR' package (Lê & Husson, 2008). Spider graphs were created using the 'radarchart' function of the 'fmsb' package (Nakazawa, 2024). Analysis of variance (ANOVA) was conducted using XLSTAT (Lumivero, Denver, CO, USA) or using R 4.3.1. In R, significance stars were generated using the 'stars.pval' function of the 'gtools' package (Warnes et al., 2023) and statistical groups were indicated by alphabetical letters determined using the 'HSD.test' function from the 'agricolae' package (de Mendiburu, 2023). Tukey's Honestly Significant Difference (HSD) analysis was also performed for multiple comparisons by using the 'HSD.test' function from the 'agricolae' package. ANOVA of sensory analysis was run with the treatment type as the independent variable.

3. Results and discussion

3.1. Sensory analysis

3.1.1. Triangle test at T03

The triangle test was performed at **T03** with 18 untrained participants. This showed that 11 of 18 panellists could distinguish between wines treated with high energy and the control ($p = 0.0144$) and 12 of 18 panellists could distinguish between the low energy treatment and the control ($p = 0.00392$), but only eight of 18 panellists could distinguish between the low energy treatment and the high-energy treatment ($p = 0.223$). Interestingly, the effect of ultrasound treatment was noted after only three months of treatment.

3.1.2. QDA at T06, T09, & T12

The QDA® method was used for **T06, T09**, and **T12** because the triangle test had shown that untrained panellists could differentiate between the ultrasound-treated wines and the control wines.

At **T06**, panellists found significant differences in overall quality between the high-energy treatment and the control ($p = 0.034$, Fisher test). Significant differences were not noted between the low energy and the control ($p = 0.632$, Fisher test) or between the high-energy treatment and the low energy treatment ($p = 0.092$, Fisher test). Panellists rated as higher quality the high-energy treatment (average rating for overall quality 6.4) over either the low energy treatment (average rating 5.8) or control (average rating 5.6). It is worth noting that the preference was related to a judgement on the overall quality and not to specific sensory descriptors. This can be seen in the spider graph (Fig. 1a).

At **T09**, panellists did not find significant differences between the treatments ($p > 0.05$), and differences were not observed in overall quality ($p = 0.941$) or overall intensity ($p = 0.198$). Significant differences were noted for 'gustatory red fruit' between the low energy treatment and control ($p = 0.038$, Fisher test) and between the low energy and high-energy treatments ($p = 0.038$, Fisher test), with the low energy treatment noted to be lower in 'gustatory red fruit' than either the control or treatment. In contrast, no difference was observed for 'gustatory red fruit' between the high-energy treatment and control ($p = 1.000$, Fisher test). As at T06, participants preferred the high-energy treatment (average rating for overall quality 6.7) over either the low energy treatment (average rating 6.3) or control (average rating 6.0), but this was not statistically significant ($p = 0.941$). These differences can also be seen in the spider graph (Fig. 1b).

At **T12**, panellists did not find significant differences between the treatments ($p > 0.05$), and differences were not observed in overall quality ($p = 0.298$) or overall intensity ($p = 0.994$). This can also be seen in the spider graph (Fig. 1c).

To recapitulate, panellists rated high-energy treatment wines at T06 and T09 as higher quality than other treatments. This seems to agree with Gavahian et al. (2022). Ahmad et al. (2023) also noted a preference for ultrasound-treated wines in both an untrained consumer panel and experienced oenologists. In contrast, Singleton and Draper (1963) noted the presence of a distinctive 'scorched' aroma in ultrasound-treated wines and consequently did not see a preference for ultrasound-treated wines. The wine style and composition and duration of treatment could likely influence the formation of such negative characters. Solar et al. (2023) noted that longer ultrasound treatment times were associated with negative effects on wine quality. Cui et al. (2011), who experimented with the use of ultrasound to end fermentation early and produce sweet, low-alcohol wines, found that treatments of 20 min or less had a positive organoleptic effect. In comparison, treatments of 30 min or more had a negative organoleptic effect on white wines. Fresno Flórez (2019) also found consumers preferred the non-ultrasound-treated wines due to the presence of atypical oxidation aromas in the ultrasound-treated young red wines (170 mins cumulative treatment of young wine on lees), which was not the case in the present experiment.

It must be noted that the wines under analysis had already been aged for five years (vintage 2017) before this experimentation. During this storage period, it can be assumed that they have reached a certain degree of chemical and sensorial equilibrium. For this reason, the effect of applying ultrasound after such a long phase of natural ageing appears far from obvious, and therefore the results are very interesting.

3.2. Wine colour (CIELab) analysis

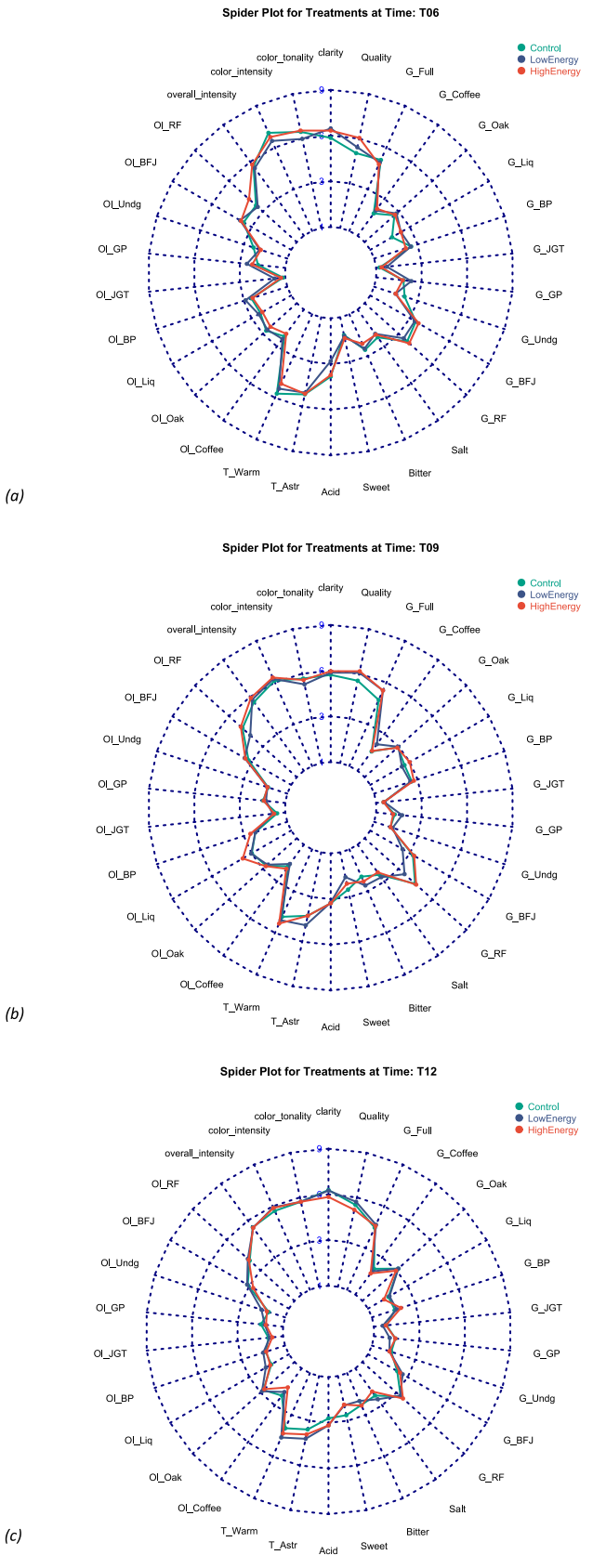
ANOVA showed that the red wine colour did not differ ($p > 0.05$) between treatments or interaction between treatments and treatment times (treatment × time) but did differ ($p \leq 0.05$) between treatment times, as is shown in Table 2. Separate analyses for each time were also not significantly different (Table S2, supporting information).

Briefly, with time the bottled wines became slightly darker (lower L^* values, Table 2), less red (lower a^* values), and less yellow (lower b^* values). They also decreased in both chroma (C^*) and hue (h). This is expected, given that red wine tends to move from purple or ruby to brick red or brown with age (McRae et al., 2012). Importantly, this process occurred independently of the treatment (Table 2 and Table S2, supporting information).

In contrast, previous studies have noted some impact of ultrasound on wine colour. Masuzawa et al. (2000) noted an increase in L^* and a decrease in both a^* and b^* in red wine matured using ultrasound. However, changes in wine colour seemed to primarily occur when ultrasound treatment was undertaken during the fermentative maceration stage, and the impact of ultrasound treatment on wine colour during the ageing phase may exert a different effect (García Martín & Sun, 2013). In contrast, Ahmad et al. (2023) found no impact on CIELab colour due to a single ultrasound treatment, in agreement with the present study.

3.3. Wine chemical analysis: basic oenological parameters

Levels of tartaric acid, malic acid, acetic acid, free sulfur dioxide, and total sulfur dioxide differed significantly between treatment times, as can be seen in Table 3. Separate analyses for each time were not significantly different (Table S3, supporting information), therefore treatments did not affect these parameters. Most importantly, neither free sulfur dioxide (Fig. 2e) nor total sulfur dioxide (Fig. 2d) varied due to treatment. This means that the treatment did not increase the risk of oxidation or microbial spoilage, which is of paramount importance to the commercial quality of the wine. As is typical in wine, these parameters varied with age. For example, free sulfur dioxide will reduce over time due to reactions with other compounds in wine such as acetaldehyde (Waterhouse et al., 2024), and organic acids such as tartaric acid



(caption on next column)

Fig. 1. Spider plots of sensory analyses; a. T06, b. T09, and c. T12. Ol_RF, olfactory red-fruit; Ol_BFJ, olfactory black fruit jam; Ol_Undg, olfactory-undergrowth; Ol_GP, olfactory green pepper; Ol_JGT, olfactory jasmine green tea; Ol_BP, olfactory black pepper; Ol_Liq, olfactory liquorice; Ol_Oak, olfactory woody; Ol_Coffee, olfactory coffee/smoky; T_Warm, taste warmness; T_Astr, taste tannicity/astringency; Acid, taste sourness; Sweet, taste sweetness; Bitter, taste bitterness; Salt, taste salty-sapidity; G_RF, gustatory red fruit; G_BFJ, gustatory black fruit jam; G_Undg, gustatory undergrowth; G_GP, gustatory green pepper; G_JGT, gustatory jasmine green tea; G_BP, gustatory blackpepper; G_Liq, gustatory liquorice; G_Oak, gustatory woody; G_Coffee, gustatory coffee/smoky; G_Full, gustatory full bodied/viscous; Quality, overall quality.

react with ethanol to form ethyl esters, decreasing the perception of acidity in aged wines (Edwards et al., 1985; Wang et al., 2015).

Tentatively, the observed trends between T0 and T3 for tartaric acid were probably due to multiple phenomena, associated with the mutual equilibria between the organic acid, their salts, their esters, and other components that contain, produce, or consume them in wine. The concentrations of the major acids in wine are not independent, as wine is a very complex buffer solution of a mix of weak acids with multiple and overlapping optimal buffering ranges (e.g. malic, tartaric, and lactic acid).

Instead, the change in acetic acid content (here determined enzymatically) might be due to the observed evolution of acetate esters (see section 3.5), in particular isopentyl acetate, that displayed a neat decrease between T0 and T3.

García Martín and Sun (2013) noted that most studies have not found any difference in pH or titratable acidity due to ultrasound treatment. One exception is that Singleton and Draper (1963) saw a decrease in volatile esters and an increase in acetic acid, while Fresno Flórez (2019) saw an increase in volatile acidity due to ultrasound treatment.

Given the key role of pH in maintaining wine microbial stability, it is beneficial that ultrasound treatment has no impact. For sulfur dioxide, the present study is also in agreement with the literature, where no difference was noted in sulfur dioxide levels due to ultrasound treatment (Cui et al., 2011; García Martín & Sun, 2013).

3.4. Wine chemical analysis: LC-MS

3.4.1. Anthocyanins at T0, T03, T09, and T12

Key anthocyanins were identified based on their LC-MS/MS signals (Table 4) and comparison with the literature (Heier et al., 2002; Ivanova Petropulos et al., 2014; Marquez et al., 2012; Mazzuca et al., 2005; McKay et al., 2015; Morata et al., 2007; Nave et al., 2010; Sánchez-Ilárduya et al., 2014; H. Wang et al., 2003), as described in the methods section. Their relative abundances in the wines changed with time (Table 4). However, there was no significant difference based on the treatment (Table 4 and Table S4).

The presence of anthocyanin complexes such as anthocyanins with catechin, vinylphenol, and aldehyde adducts, such as vitisins and portosins, is typical for aged wines(Brouillard et al., 2003; Mateus & de Freitas, 2001; H. Wang et al., 2003). The presence of anthocyanin-acetate complexes is also interesting, and the formation of these stable pigments may be related to the decrease in acetic acid. Reactions can also occur between acetaldehyde (produced by the oxidation of ethanol), tannins, and anthocyanins, also forming more stable compounds (Es-Safi et al., 2008; Romero & Bakker, 2000; Waterhouse et al., 2024). In contrast, organic acids, such as tartaric, acetic, and malic acid, have been shown to have minimal impact on the formation of anthocyanin complexes (Romero & Bakker, 2000).

A PCA of all anthocyanins and associated compounds (filtered using ANOVA) was conducted (Fig. 3) to investigate the effects of the treatments on the colour development during the storage. In this analysis, trends are only discernible at T03 and T09, and an overall PCA does not show such trends given the confounding effect of time. However, the effect is particularly noticeable at T09 (see also section 3.4.3), where

Table 2
Table of p values for CIELab by time, treatment, and interaction (treatment × time); '***' $p = 0$ - 0.001, '**' $p = 0.001$ - 0.01, '*' $p = 0.01$ - 0.05, n.s. $p = 0.05$ – 1.0 (not significant).

CIELab parameters	Treatment (p value)	Time (p value)	Interaction (p value)
L^*	0.6931 (ns)	0.0002037 ***	0.5848 (ns)
a^*	0.8888 (ns)	1.871e-05 ***	0.8107 (ns)
b^*	0.6931 (ns)	0.0002037 ***	0.5848 (ns)
C^*	0.8821 (ns)	8.848e-05 ***	0.8186 (ns)
h	0.6755 (ns)	2.542e-12 ***	0.1755 (ns)

Table 3
Table of p values for basic oenological parameters by time, treatment, and interaction (treatment × time); '***' $p = 0$ - 0.001, '**' $p = 0.001$ - 0.01, '*' $p = 0.01$ - 0.05, n.s. $p = 0.05$ – 1.0 (not significant).

Miura parameters	Treatment (p value)	Time (p value)	Interaction (p value)
Tartaric acid	0.5886 (ns)	5.676e-14 ***	0.006311 **
Malic acid	0.4604 (ns)	5.382e-23 ***	2.699e-05 ***
Acetic acid	0.9893 (ns)	1.563e-49 ***	4.571e-05 ***
SO_2 total	0.9082 (ns)	1.109e-12 ***	0.6118 (ns)
SO_2 free	0.8592 (ns)	5.099e-32 ***	2.477e-08 ***

PC1 separates the treatments. At T03, the treatments are separated by PC1 and PC2, but the control and high-energy treatments clustered on PC1. It appeared, however, that any effects on anthocyanins are counteracted by T12, suggesting that additional treatment has a detrimental effect.

Results in the literature regarding the effect of ultrasound on anthocyanins during wine ageing are controversial. In agreement with this study on an aged red wine, Ferraretto and Celotti (2016) found no influence on free anthocyanins due to ultrasound treatment in young red wines. In contrast to this study, Masuzawa et al. (2000) noted an increase in anthocyanin levels with ultrasound treatment of a young red wine, while Lukić et al. (2019) and Fresno Flórez (2019) found a decrease in anthocyanin levels of young red wines.

Lukić et al. (2019) attributed this to the opening of the pyrylium ring in anthocyanins because of the formation of free radicals due to cavitation, causing the anthocyanins to form chalcones. Lukić et al. (2019) found that the key factor influencing anthocyanin levels was the ultrasound frequency used, which was not varied in the present study. Sun et al. (2019) found that ultrasound treatment favoured the conversion of cyanidin-3-O-glucoside to methylpyranocyanidin-3-O-glucoside.

In contrast, Fresno Flórez (2019) found lower levels of all pigments, including pigments formed by the acylation of glucose with acetic and coumaric acids, and stable anthocyanin complexes such as vitisin A and B.

3.4.2. *Polyphenols at T0, T03, T06, T09, and T12*

PCA (filtered using ANOVA for polyphenols vs. treatment time) was again used. The results of the analysis of polyphenols presented no specific trends related to the treatments (Fig. 4). Only one compound (DAD 280 nm, RT 15.5 mins, $p = 0.04552$ at T03, m/z 570.1) was found to be statistically significant for treatment, but the interpretation of this evidence is questionable. This trend would initially seem surprising, given that the polymerisation of polyphenols is a key factor in wine ageing. However, the wines being studied had already been aged for 5 years before the start of the experiment, and existing literature on the effect of ultrasounds is restricted to studies using young wines.

Ahmad et al. (2023) found that ultrasound treatment of young red wines had a significant influence on total polyphenols, with ultrasound either increasing or decreasing the total polyphenols depending on the duration of treatment. Ferraretto and Celotti (2016) also noted significant differences in polyphenol composition of young red wines due to ultrasound treatment, with wines treated using ultrasound having higher catechins, and they suggested that the treatment may promote the depolymerisation of monomeric catechins from complexes.

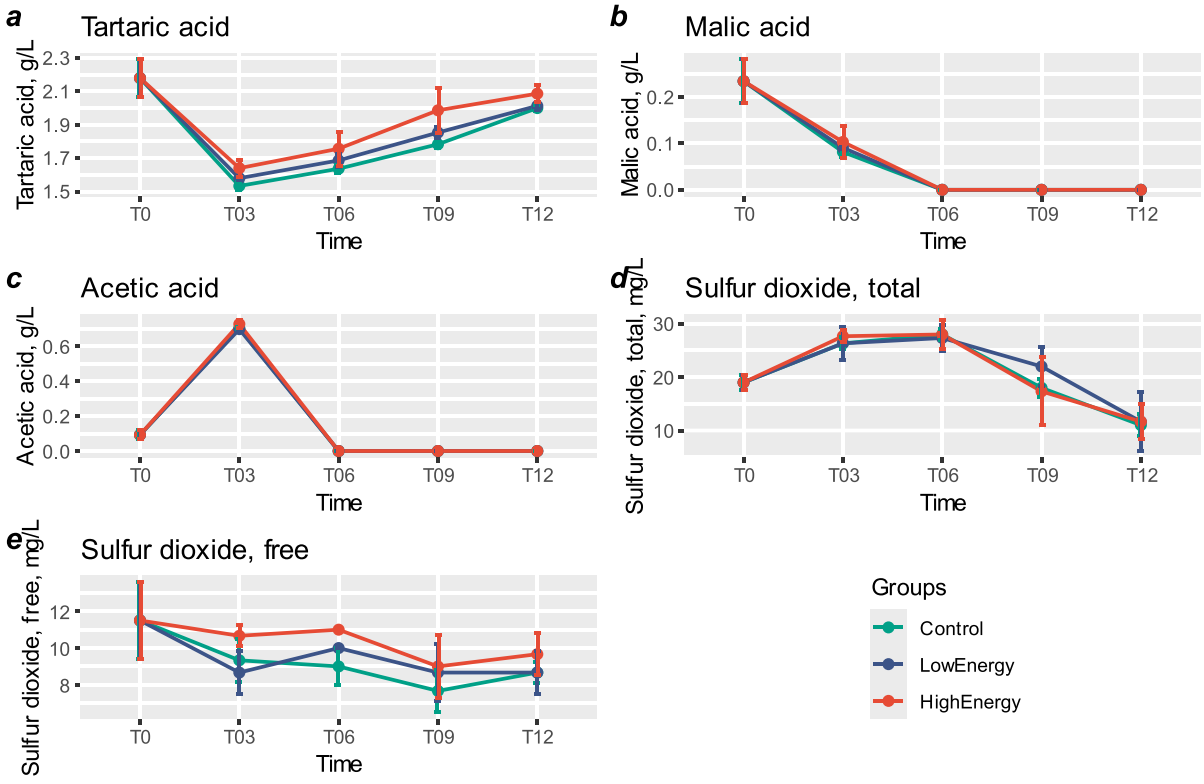


Fig. 2. Changes in levels of organic acids and sulfur dioxide over the experimental period; a, tartaric acid, b. malic acid, c. acetic acid, d. total sulfur dioxide, e. free sulfur dioxide. Error bars indicate ± 1 standard deviation.

Table 4
Table of p values for anthocyanins by time, treatment, and interaction (treatment × time); '***' $p = 0$ - 0.001, '**' $p = 0.001$ - 0.01, '*' $p = 0.01$ - 0.05, n.s. $p = 0.05$ – 1.0 (not significant).

Anthocyanin	Reference	Treatment p value	Time p value	Interaction p value	Retention time (mins)	*m/z*	MS/MS fragments
petunidin-3-O-glucoside	(Mazzuca et al., 2005; H. Wang et al., 2003)	0.6679	5.195e-09 ***	0.179	8.52	479.2	316.9, 301.9, 273.9
peonidin-3-O-glucoside	(Mazzuca et al., 2005; H. Wang et al., 2003)	0.9323	0.0001457 ***	0.8846	9.83	463.2	301.0, 286.2
malvidin-3-O-glucoside	(Mazzuca et al., 2005; H. Wang et al., 2003)	0.6044	4.57e-06 ***	0.2439	10.56	493.2	331.0, 315.1
delphinidin glucuronide (†)	(McKay et al., 2015)	0.5243	7.149e-07 ***	0.1155	11.37	479.2	303.0, 112.9, 85.0
vitisin A	(Marquez et al., 2012; Morata et al., 2007)	0.8826	2.276e-14 ***	0.08632.	11.47	561.2	399.1
vitisin B	(Marquez et al., 2012; Morata et al., 2007)	0.9531	3.814e-14 ***	0.5801	12.27	517.2	355.2
cyanidin-3-O-(6-O-acetyl) glucoside	(Heier et al., 2002; Marquez et al., 2012)	0.98	2.63e-17 ***	0.6581	12.90	491.0	287.0
cyanidin-3-p-(6-O-p-coumaryl))-glucoside	(Heier et al., 2002)	0.2594	0.004311 **	0.05331.	14.56	595.3	355.1
p-coumaroyl-vitisin A	(Ivanova Petropulos et al., 2014)	0.9977	2.216e-18 ***	0.9353	14.75	707.3	399.1
malvidin-3-O-(6-O-acetyl) glucoside	(Mazzuca et al., 2005)	0.8152	1.35e-07 ***	0.5493	14.85	535.2	331.0
delphinidin-3-O-(6-O-acetyl) glucoside	(Mazzuca et al., 2005)	0.3179	4.582e-05 ***	0.03373 *	15.16	507.2	
malvidin-3-glucoside-4-vinyl-catechin	(Sánchez-Ilárduya et al., 2014)	0.9695	4.77e-20 ***	0.4173	16.05	805.4	643.1, 491.1, 387.1
petunidin-3-O-(6-O-p-coumaryl)glucoside	(Mazzuca et al., 2005)	0.8812	5.592e-20 ***	0.008827 **	16.88	625.2	463.0
peonidin-3-O-(6-O-p-coumaryl)-glucoside	(Mazzuca et al., 2005)	0.9356	1.262e-13 ***	0.5366	17.81	609.2	447.0
malvidin-3-O-(6-O-p-coumaryl)glucoside	(Mazzuca et al., 2005)	0.9632	1.372e-24 ***	0.07211.	17.90	639.2	477.0, 462.1

(†) tentative assignment.

However, as with Ahmad et al. (2023), this was dependent on the treatment intensity and time.

3.4.3. *MFA of polyphenols, anthocyanins, and PAC at T09*

The data from T09 suggest that the wines are distinct in terms of analytical parameters. An MFA of these data (anthocyanins, phenols, and proanthocyanidins PAC, filtered using ANOVA) showed clear separation (Fig. 5a). In the MFA loadings (Fig. 5b), the first PAC Principal Component (PC1) had a strong negative effect on F1 and a strong positive effect on F2, while the second PAC Principal Component had a strong negative influence on both F1 and F2. The first anthocyanin component had a strong positive effect on both F1 and a moderate positive effect on F2, and the second anthocyanin Principal Component had a strong negative effect on F1 and a moderate positive effect on F2. The first phenolics component had a strong positive effect on F1 and a slight positive effect on F2, and the second phenolics component had a slight negative effect on F1 and a strong positive effect on F2. This means that the low-energy treatments were separated by PAC on F1 and polyphenols on F2, the high-energy treatment by PAC on F2, and the control by anthocyanins and polyphenols on F1. This reinforces the importance of PAC and polyphenols to distinguishing the treatments at T09.

Solar et al. (2023) noted that the reactions seen in ultrasound-treated wines are similar to those seen in wine during oak ageing.

3.4.4. *PAC at T12*

Oligomeric condensed tannin data were analysed at T09 (section 3.4.3) and T12. ANOVA was used to filter the data, and only three variables were found to be significant. These were graphed using PCA (Fig. 5c and d). There is clear separation between the control and the two treatments on PC1, and some separation between the two treatments on PC1 and PC2. This shows that there is clear separation between the treatments and the control. However, the separation between the two treatments was not as clear, suggesting that the high-energy treatment offers little difference.

This is in agreement with Xue et al. (2023), who found that flavan-3-ols polymerised via acetaldehyde and glycolic acid bridges in model wine that had been treated with ultrasound. Ferraretto and Celotti (2016) found higher levels of PAC in wines treated with ultrasound without lees, but lower levels in wines treated with ultrasound and with lees. In contrast, Ahmad et al. (2023) noted lower levels of tannins in ultrasound-treated young red wines, but suggested this might be due to depolymerisation as a result of acoustic cavitation.

3.5. *Volatile compounds determined with GC×GC–MS*

As one might expect, the effects of the treatments on the fingerprint of volatile compounds evaluated using GC×GC–MS analysis were quite distinct from the patterns shown in the LC-MS analysis of anthocyanins.

Again, ANOVA (analyses of overall data and on each time separately, any variable that was significant in one or more analyses was preserved) was used to filter the data prior to PCA (Fig. 6). Patterns are hard to distinguish, though the high-energy treatment is separated on PC1 for T03 (Fig. 6c), and the two treatment levels are somewhat separated from the control on PC2 for T09 (Fig. 6e). The significant compounds used for the PCA, along with their retention times and *m/z*, are listed in Table 5.

Significant differences in isopentyl acetate ($p = 0.04905$) and trans-β-damascenone ($p = 0.04751$) were noted across all times. Isopentyl acetate (isoamyl acetate) is described as having banana, estery, fruity, ripe, solvent, and sweet aromas, as well as banana, fruity, green, ripe, sweet flavours (Luebke, 1980–2021). trans-β-damascenone (E-β-damascenone) is described as having berry, blackcurrant, floral, fruity, honey, minty, plum, rose, tobacco, and woody aromas, as well as berry, floral, fruity, green, herbal, honey, jammy, minty, plum, tobacco, and tropical flavours (Luebke, 1980–2021). These changes may have been reflected in the changes in sweetness ($p = 0.0007$) and in red berry

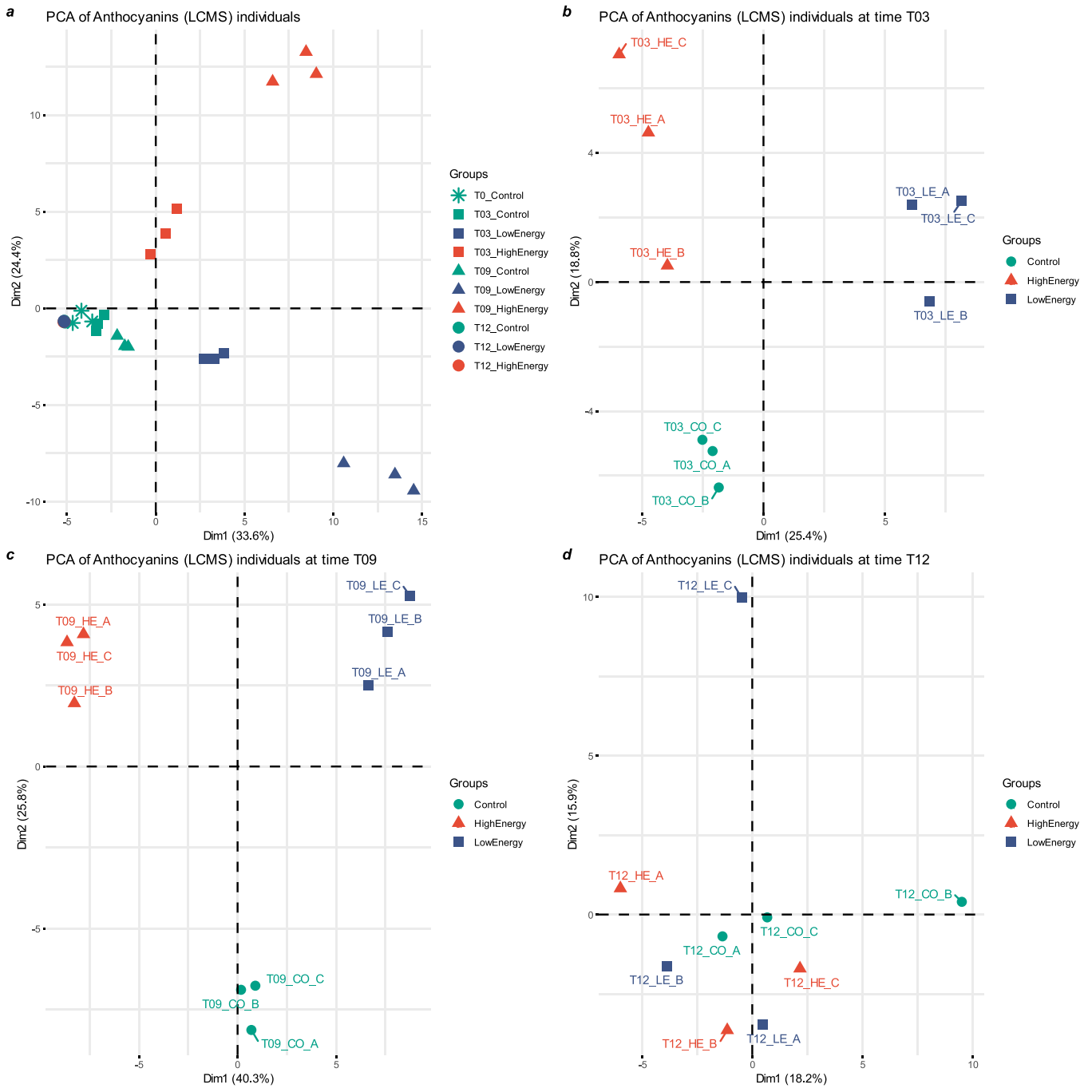


Fig. 3. PCA of anthocyanins; a. overall (all times), b. PCA at T03, c. PCA at T09, d. PCA at T12.

aromas ($p = 0.0003$) and flavours ($p = 0.002$) that were significant with time but not with treatment.

Changes in aromas responsible for spice and oak flavour such as 4-ethylguaiacol ($p = 9.575\times10^{-08}$ comparing T0 and T12) and (*cis*)-oak-lactone (cis-β-methyl-γ-octalactone; $p = 1.783\times10^{-09}$ comparing T0 and T12) are also noticeable with time. Such changes are typical of wine ageing.

4. Conclusions

The study aimed to determine the utility of ultrasound as a technology to improve and eventually accelerate the ageing of bottled wine for technology transfer to wineries. The main limitation of this study was that differences due to ultrasound might not be as clear cut as would be observed in a younger wine and that existing bottle variation (not uncommon in aged wines (Robinson & Harding, 2019)) might influence the sensory analysis.

Overall, ultrasounds have a measurable impact on sensory and chemical characteristics of wine. The sensory analysis results at 6 months of treatment suggest clear differences in panellist rating of overall quality between the high-energy treatments and the control.

These differences were also noted in the chemical profile of the wine, although the picture is not straightforward. The anthocyanin profile was distinctive after three months and nine months of ultrasound treatment but not after 12 months of treatment, suggesting that prolonged treatment may not be beneficial. These distinctions were also seen in an MFA of all LC-MS variables at nine months, and for PAC at 12 months. In contrast, the impact on the volatile profile was most visible at three

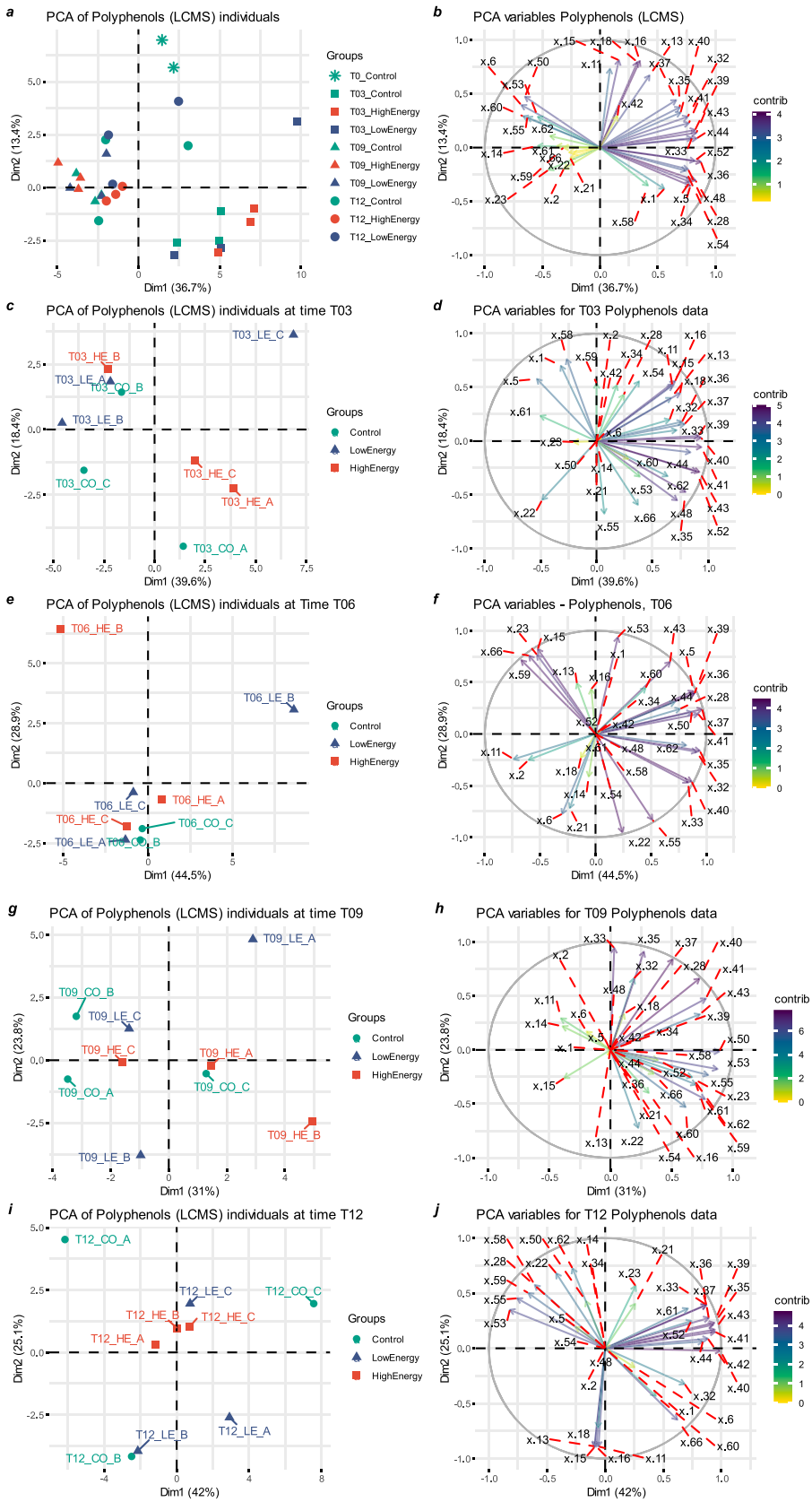


Fig. 4. PCA of polyphenols; a. & b. overall (all times), c. & d. PCA at T03, e. & f. PCA at T06, g. & h. PCA at T09, i. & j. PCA at T12. Variables are coloured based on their contribution ('contrib') to the PCA.

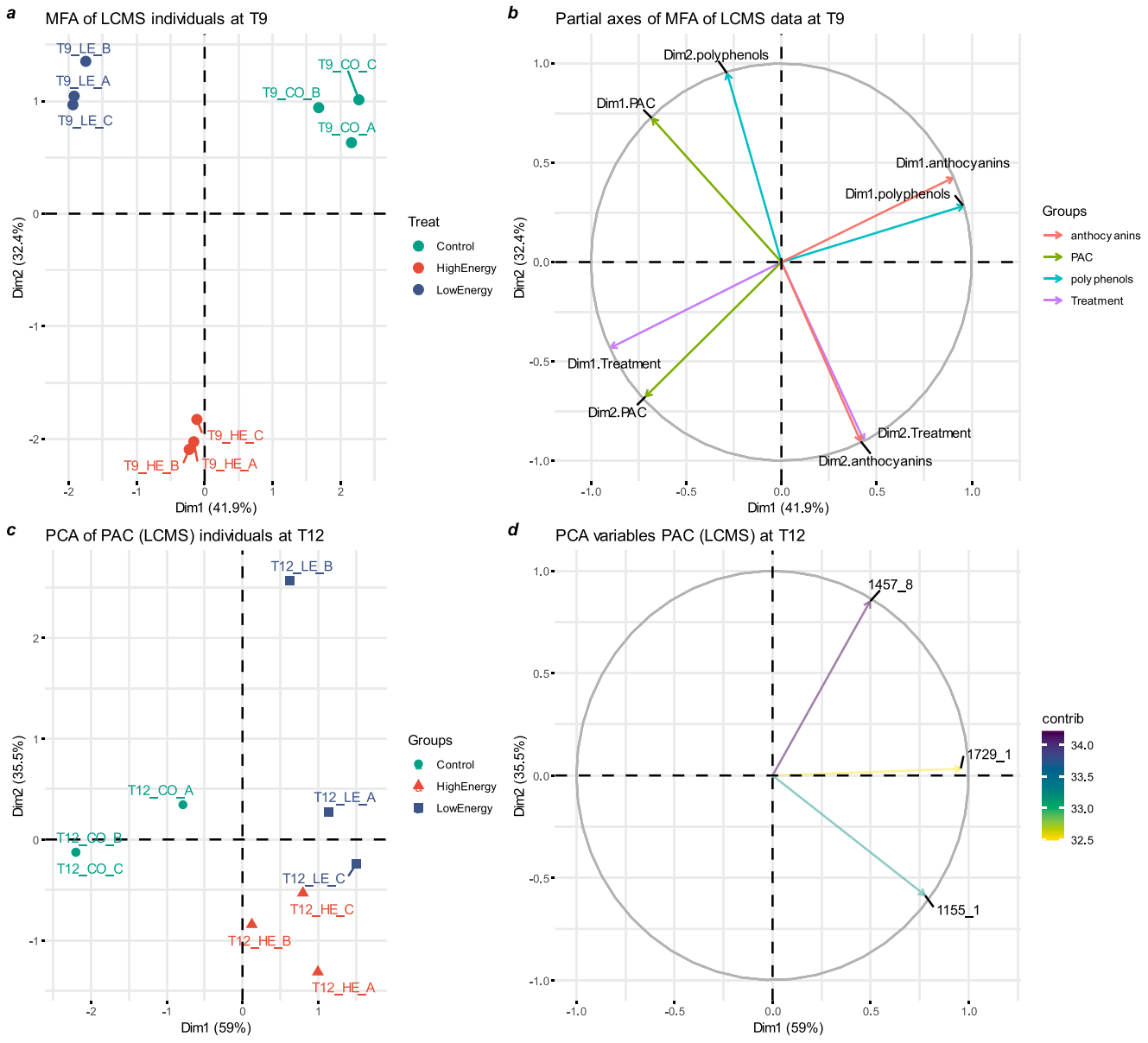


Fig. 5. Multifactorial analysis of LC-MS data; a. MFA of anthocyanins, phenols, and PAC at T09, b. MFA partial axis plot; c. PCA of PAC at T12, d. loadings plot. Variables are coloured based on their contribution ('contrib') to the PCA.

months. The polyphenol profile did not show any specific trends, and only one compound differed significantly due to the treatment. This is likely because the wine had aged for five years prior to treatment and was likely already at an equilibrium.

The differences observed in the volatile profile with age also influenced the sensory profile, with differences in isopentyl acetate and trans–β-damascenone likely influencing the sweetness and the red berry aromas and flavours of the wine.

At 6 months, panellists rated the high-energy treatment as higher in overall quality, suggesting that the treatment improved the quality of the wine. Importantly, free sulfur dioxide levels were not influenced by the ultrasound treatment, meaning that the wines were still protected from oxidation and spoilage microorganisms.

In summary, from a business strategy point of view, since consumer perception of wine is crucial to ensure sales and customer satisfaction, it is suggested that six months may be the best treatment time. Although high-energy treatment was found to be the best, since the difference between high-energy and low-energy treatment is minimal, low energy treatment might be more suitable given the lower costs. Future studies should examine the application of ultrasounds to young red wines (i.e., wines that presumably not yet reached a chemical equilibrium). In contrast, the results of this study refer to wines already aged for a few years in a bottle.

Declarations

Ethics statement

Participants gave informed consent, and an affirmative reply was required to enter the survey. They were able to withdraw from the survey at any time without giving a reason and without any disadvantage to themselves. The privacy rights of all subjects were observed. The panellists were all regular wine consumers recruited voluntarily from the Free University of Bozen-Bolzano and NOI Techpark (Bolzano, Italy), and were not compensated. For QDA, panellists were given a maximum of nine 30-mL wine samples plus one 30 mL calibration sample per sensory session (equivalent to 39.3 mL of pure ethanol) and were encouraged to taste the sample without swallowing. The products tested

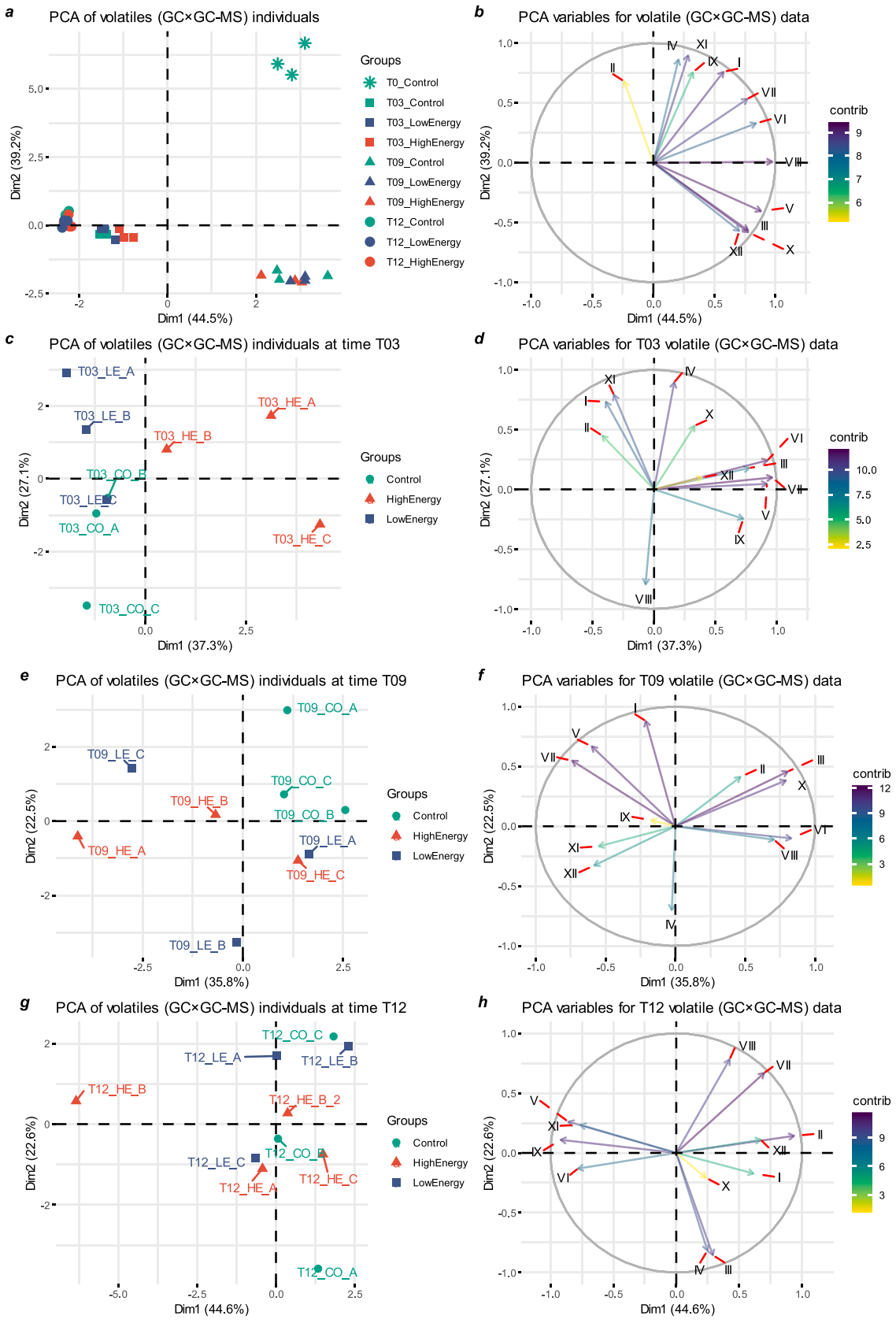


Fig. 6. PCA of volatile compounds; a. individuals for all times, b. variables for all times, c. individuals for T03, d. variables for T03, e. individuals for T09, f. variables for T09, g. individuals for T12, h. variables for T12. Codes are as per Table 5. Variables are coloured based on their contribution ('contrib') to the PCA. Compound label codes are as per Table 5.

Table 5
GC×GC compounds with retention times and relative masses.

Code	Compound	RT1 (min)	RT2 (*sec*)	Base mass	MS/MS fragments
I	isopentyl acetate (1-butanol, 3-methyl-, acetate)	12.1	0.140	43.02	70.07, 55.06, 39.03
II	(2S,3S)-(+)−2,3-butanediol	22.1	0.646	45.04	55.10, 43.02,
III	2,4-di-tert-butylphenol	55.6	1.09	191.13	65.04, 57.07, 53.05, 51.03, 41.05
IV	trans-β-damascenone	40.9	1.19	69.03	65.03, 52.30, 43.98, 41.06
V	β-ionone (4-(2,6,6-Trimethyl-1-cyclohexenyl)−3-buten-2-one)	29.7	1.70	93.05	65.05, 55.06, 43.04, 41.06
VI	ethyl palmitate	54.6	1.28	88.04	61.03, 55.05, 43.06
VII	1,1,6-Trimethyl-1,2-dihydronaphthalene	37.9	0.592	157.09	63.00, 56.62, 51.85, 44.00, 33.98
VIII	n-decanoic acid	55.1	1.05	60.02	56.10, 41.05
IX	1,1′-oxybisoctane	38.3	0.0399	57.07	43.06

were safe for consumption, the study was conducted following the Declaration of Helsinki (World Medical Association, 2013), and all procedures were performed in compliance with relevant laws and institutional guidelines.

Ethical statement

The authors confirm that the appropriate protocols for protecting the rights and privacy of all participants were utilized during the execution of the research, including no coercion to participate, full disclosure of study requirements and risks, consent of participants, no release of participant data without their knowledge, and the ability to withdraw from the study at any time.

CRediT authorship contribution statement

Gavin Duley: Writing – review & editing, Writing – original draft, Investigation, Formal analysis, Data curation. **Simone Poggesi:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Lorenzo Longhi:** Writing – review & editing, Formal analysis. **Edoardo Longo:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Conceptualization. **Emanuele Boselli:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors have no conflicts of interest to report.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.afres.2024.100540.

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