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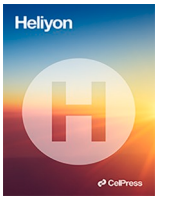

Research article

Red wine maceration with grapevine-cane residues: Influence of format and toasting level

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ABSTRACT

Grape production generates a significant amount of pruning waste that could be repurposed for different applications. Recently, the use of pruning canes as potential additives to enhance wine quality has been proposed and studied, but further research is needed to better understand their effects. Therefore, the aim of this study was to characterize some of the physicochemical properties of Cabernet Sauvignon grapevine canes, and to assess the impact of their maceration in red wine, using different formats (i.e., powder, granules, and discs) and toasting levels (i.e., low and high). The results show that the canes analyzed are rich in phenolics such as *trans*-ε-viniferin and *trans*-resveratrol (i.e., 30.1 ± 0.2 and 5.3 ± 0.1 mg/g extract respectively), and that their maceration in red wine, produced variable outcomes depending mainly on the format employed. For instance, treatments with powder vine canes led to a reduction in total phenolics (−19.4 %), antioxidant capacity (−14.9 %), total anthocyanins (−19.5 %), and catechins (−9.5 %), compared to the untreated control. Instead, granules and discs produced no significant variation when compared to untreated or oak treated samples, after 24 days of maceration. Given that the cane format employed significantly influences the phenolic composition of the wines, future studies could explore the mechanisms behind these differing effects, as well as the sensorial implications of these changes.

1. Introduction

With around 80 million tons harvested annually and a surface area of over 7.3 million hectares worldwide, including vineyards for all uses, grapes are one of the most extensively cultivated fruit crops [1]. This extensive production generates significant vineyard wastes, especially pruning canes, which are considered their major residue, with an average production ranging from 2 to 5 tons per hectare each year [2,3]. Pruning activities have a crucial role in grape production, impacting vine form, size, yield, and quality [4].

Depending on their sanitary condition, vine pruning canes may be chipped on-site and used as mulch, converted into compost material, disposed around the vineyard [5,6], or even burned in the field, leading to air pollution emissions [7]. However, grapevine

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canes could be re-used for various value-added applications [3,8], due to their content of bioactive compounds, including minerals, vitamins, phenolic compounds (i.e., stilbenes), and other substances [4,9–12].

The structural components of grapevine pruning canes represent about 80 % of their total dried matter [13], having important variations depending on the grape variety. For instance, the compositional ranges of cellulose, hemicellulose, and lignin have been reported to vary between 31.9 and 41.1 %, 21.7–28.3 %, and 14.8–39.2 %, respectively [14]. Cellulose consists of unbranched and long glucose polymers, lignin is composed of aromatic rings with various branches, and hemicellulose appears as a random and amorphous structure combining various saccharides (i.e., xylose, mannose, glucose, galactose, etc.). Some of these constituents can be degraded into volatiles during thermal treatments or combustion [15].

Vine pruning residues could be repurposed as raw materials for biofuels, the production of activated carbon or other industrial materials [14], and the extraction of valuable bioactive compounds [10,11,16]. Like so, recent studies focusing on the revalorization of vine canes have reported their use as potential alternatives to oak treatment aids during the aging of wines [19–25]. In this regard, it has been demonstrated that toasting conditions play a significant role in the release of valuable compounds from the vine canes to the wine [17–19]. Like so, other aspects such as wood format or vine cultivar have also been shown to produce different outcomes when treating wines with vine canes [19–25].

In this context, the aims of this study were to characterize the physico-chemical features of Cabernet Sauvignon grapevine canes, focusing on their thermal stability and phenolics composition, and to evaluate the influence of different cane formats (i.e., powder, granules, or discs) and toasting levels, on the phenolic content of wine-like model solutions and red wine treated with them.

2. Materials and methods

2.1. Physico-chemical features of grapevine canes

2.1.1. Plant material

Twelve sample packs of ca. 50 Cabernet Sauvignon grapevine canes each (50 cm × 5 mm, length x diameter, approx.) were sourced from Univiveros nursery in Paine, Chile. The plant material was collected during the winter pruning activities of 2021 from different sections of a varietal garden, from June to August, and dried at room temperature for one year. Thereafter, the grapevine canes were cut into smaller pieces (2–4 mm length) to facilitate their grinding (Oster BLSTKAG-NPB-052, México) and fractionation according to size (Fig. S1). The ground material was fractionated according to the particle size, using two stainless-steel laboratory sieves (0.45 and 2 mm), and the sieved materials were analyzed to know their thermal stability and their elemental composition, as described below.

2.1.2. Thermal stability

To determine the thermal stability of the Cabernet Sauvignon grapevine canes, a thermogravimetric analysis (TGA) was performed. Aside from continuously recording time, temperature, and weight, the first derivative of such recorded data, known as derivative thermogravimetry (DTG) was obtained [26]. TGA and DTG were carried out according to Castro and Morales-Quintana (2019) [27], using an STD 650 Thermal Analyzer (Waters TM, TA Instrument, USA). For this purpose, 10 mg of powdered vine canes (particles <0.45 mm) were deposited onto a platinum crucible. Considering the wide temperature range of degradation that the different components of wood expose [15], the samples were heated from 50 to 600 °C at a continuous rate of 10 °C/min, using air as a reactive gas at a mass flow of 50 mL/min. Nitrogen was employed in the electronic balance (50 mL/min) as a protective gas.

2.1.3. Preparation and analysis of grapevine cane extracts

Ground and sieved cane particles (<2 mm) were weighted (2.5 g) and placed into 100 mL Erlenmeyer flasks filled with 25 mL of 80 % ethanol (in triplicate). The extraction process was assisted with an ultrasonic water bath in which the samples were placed for 10 min (25 ± 2 °C; 53 KHz; 100 % Intensity). The liquid extracts obtained were filtered (Whatman No. 42; Merck, Germany) and dried in a benchtop vacuum concentrator (CentriVap, Labconco Corporation, USA). The dried extracts obtained were stored at −40 °C until analyzed for total phenolics (TPC), low-molecular-weight phenolics (HPLC-DAD-FLD), and antioxidant activity (DPPH). For this purpose, a stock solution of the extracts was prepared in methanol (1 mg/mL) and analyzed as described in the following sections.

2.2. Maceration experiments using grapevine canes

2.2.1. Plant material processing: fractionation and toasting process

Grapevine canes from Cabernet Sauvignon were used for maceration experiments using wine-like model solutions and red wines. As mentioned in section 2.1.1, the plant material was manually cut into pieces of 2–4 mm in length, ground, and sieved. Thus, three different cane formats were obtained, identified as a) powder (particles <2 mm), b) granules (particles >2 mm), and c) discs (2–4 mm in length). According to the results of the thermal analysis, the cane materials were toasted at 180 °C using a universal convection stove (UNE-550, Memmert, Germany), during two different times (i.e., 44 and 120 min), following the directions of Cebrián-Tarancón et al. (2019) [21], and Chira & Teissedre (2013) [28], respectively. Consequently, three different toasting levels were obtained: i) Untoasted wood, ii) Low toast (45 min at 180 °C), and iii) High toast level (2 h at 180 °C).

2.2.2. Maceration of grapevine canes in wine-like model solution

To study the evolution of the total phenolic compounds obtained from the maceration of grapevine-cane materials over time, an initial experiment was performed using wine-like model solutions. The experimental conditions suggested by Cebrián-Tarancón et al. (2019) [21] were followed, with slight modifications. Briefly, the model solutions were composed by distilled water (87.5 %, v/v), ethanol (12.5 %, v/v), tartaric acid (5 g/L), and adjusted to pH 3.5 with NaOH 1 N. A wood dose of 12 g/L was used and a total maceration time of 36 days. The experiments were performed in 50 mL screw cap tubes, including the three wood formats (i.e., powder, granules, and discs) and toasting levels (i.e., untoasted, low, and high-level toast) as described before. Additionally, two controls treatments were also prepared, consisting of a wine-like model solutions without wood (negative control), and a control with commercial American oak wood chips (2–4 mm in size) (ViniBlock, TN, Chile). All treatments and controls were tested in triplicate. All treatments and their respective controls were kept at room temperature (20 ± 2 °C) in the dark, and gently shaken daily. After 36 days of maceration, the samples were filtered (Whatman No. 42; Merck, Germany) to remove the solid components, and the solutions obtained were refiltered using syringe filters (PTFE, 0.45 μm) before the spectrophotometric and enological analyzes. To check the evolution of the total phenolic content of the wine-like model solutions over time, and their antioxidant activity, aliquots of 2 mL from each tube were taken, filtered (PTFE, 0.45 μm), and analyzed, at 12 and 24 days of maceration, as well.

2.2.3. Maceration of grapevine canes using red wine

Based on the results of the previous experiment, using wine-like model solutions, a second experiment was performed, using a Cabernet Sauvignon red wine. Briefly, the same wood formats were tested (i.e., powder, granules, and discs), but only in their toasted variants (low and high-level toast). The same wood dose was used (12 g/L), including an oak control and a woodless control. The experiments were performed in 100 mL screw-capped dark glass bottles in triplicate. The samples were kept at room temperature (20 ± 2 °C) in the dark, and gently shaken daily. The samples were filtered at 24 days of maceration (Whatman No. 42; Merck, Germany) to remove the solid wood components. Then, the obtained wines were filtered using syringe filters (PTFE, 0.45 μm) to perform the physicochemical and enological analyzes described in the following section.

An additional transparent glass bottle (250 mL) for each of the treatments were also prepared in order to perform oxygen measurements as further detailed below. These bottles were kept under the same conditions as the rest of the bottles (see section 2.6).

2.3. Spectrophotometric analyzes of wine-like model solutions and red wines

All spectrophotometric analyzes performed were measured in a Synergy HTX multi-mode reader equipment (Biotek Instruments, USA), as explained bellow.

2.3.1. Total phenolics content

Total phenolics were determined by the Folin-Ciocalteu micro assay [29]. For this purpose, 20 μL of each sample was diluted with 1.58 mL of distilled water and then, mixed with 100 μL of the Folin-Ciocalteu reagent. Samples were pre-incubated (30 s–8 min) and then, 300 μL of sodium carbonate (20 % w/v) were added. After 30 min at 40 °C, the absorbance was measured at 765 nm. Results were expressed as mg of gallic acid equivalents (GAE) per liter, according to the respective calibration curve (50–500 mg GAE/L). In the case of red wines, the samples were diluted in distilled water (1/10) prior to the analysis. Wine-like model solutions were analyzed without dilution.

2.3.2. Antioxidant capacity

The scavenging activity against the 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical was measured according to the method adapted from Brand-Williams et al. (1995) [30]. Briefly, 75 μL aliquot of wine were mixed with 150 μL of DPPH in methanol (20 mg/L). The mixture was stirred, incubated for 10 min at 25 °C, and then, read at a wavelength of 515 nm. For the analysis of wood extracts, different extract dilutions were tested (1–250 μg/mL in methanol), and then, the half-maximal inhibitory concentration (SC_{50}, mg/L) was calculated by linear regression analysis and expressed as the mean of three determinations. On the other hand, a 1/2 dilution was made in case of wine-like model solutions, and a dilution 1/100 for red wines. The reduction of DPPH was tested in triplicate for each wine sample and the results were expressed as mg of Trolox equivalents/L, according to Cebrián-Tarancón et al. (2019) [20].

2.3.3. Methylcellulose precipitable assay

Tannin concentration was determined using the methylcellulose precipitable (MCP) assay [31]. Briefly, 25 μL of each sample were placed in 1.5 mL tubes, mixed softly with 300 μL of a fresh methylcellulose solution (0.04 % in distilled water) and incubated during 2–3 min. Then, 200 μL of a saturated solution of ammonium sulphate, and 475 μL of distilled water were added, mixed in a Vortex apparatus (MX-F, DLAB, China), and incubated at room temperature during 10 min. The samples were centrifuged at 10000 rpm by 5 min (Centrifuge 5415R, Eppendorf, Germany). Finally, 300 μL of each supernatant were placed in UV microplates (Code 782614, Brand, Germany) and read at 280 nm. The same procedure was performed for the control sample but replacing the methylcellulose polymer solution by distilled water. Then, the subtraction of both absorbances was expressed in catechin equivalents (CE), using a calibration curve (1–250 mg CE/L), and multiplied by the dilution factor (FD = 40) [31].

2.3.4. Polymeric pigments

Small and large polymeric pigments (SPP and LPP) were measured according to Harbertson et al. (2003) [32], with some modifications. Briefly, the wine samples were diluted (1:3) in a buffer (buffer 1) composed by potassium bitartrate (5 g/L), ethanol (12 %), and adjusted to pH 3.3 with hydrochloric acid (0.1 N). Two aliquots of 500 μL of the diluted samples were placed in two centrifuge tubes, respectively. The first one, was mixed with 1 mL of a buffer solution (buffer 2) containing 200 mM of glacial acetic acid, 170 mM sodium chloride, and adjusted to pH 4.9 with sodium hydroxide (0.1 N). This mixture was incubated during 10 min (25 °C). Then, 1 mL of this solution was mixed with 80 μL of a bleaching solution (potassium disulfite 0.36 M), incubated by 10 min, and read at 520 nm, using buffer 2 as blank (Abs_A). The second aliquot of diluted wine (500 μL) was mixed with 1 mL of a protein solution (1 mg/mL of bovine serum albumin dissolved in buffer 2), and incubated during 15 min. After this time, 1 mL of the supernatant was transferred to another tube, mixed with 80 μL of the bleaching solution, incubated by 10 min, and read at 520 nm (Abs_B). Then, Equations (1) and (2) were applied to obtain the index of large polymeric pigments (LPP), and small polymeric pigments (SPP), respectively [33], as indicated bellows:

$$(1.08) \times (\text{Dilution Factor}) \times (4 / 3) \times (Abs_A - Abs_B) \quad \text{Eq. 1}$$

$$(1.08) \times (\text{Dilution Factor}) \times (10 / 7) \times (Abs_B) \quad \text{Eq. 2}$$

2.3.5. Color analysis

CIELab parameters were calculated as summarized in Guzmán-Alfeo (2018) [34] with slight modifications. Aliquots of 300 μL of the obtained wines (dilution factor = 10) were placed in UV–Vis microplates (Code 781602, Brand, Germany) and read at 450, 520, an 570 nm. Similarly, wine samples were read at a wavelength of 630 nm, and the obtained absorbances were entered into the software MSCV (University of La Rioja, Spain) (https://www.unirioja.es/color/descargas.shtml) to obtain the respective color parameters. Color difference (ΔE^*_{ab}) between the red wine samples was calculated as the Euclidean distance between two points in the *L*, a*, b** three-dimensional space, as reported by Fanzone et al. (2021) [25].

Wine hue (or tonality) was calculated as the ratio of the absorption of light between 420 nm (violet) and 520 nm (green) [34].

2.4. General enological parameters of wines macerated with grapevine-cane materials

Enological parameters of the wines were determined on a Y15 automatic wine analyzer (Biosystems, Spain), according to the manufacturer's instructions, including total and free sulfite, L-malic acid, total catechins, and total anthocyanins. Other additional parameters were measured in the Cabernet Sauvignon wine at the beginning of the assay (time = 0 days), which are described in Table 1. On the other hand, the pH of the samples was checked at the beginning, and at the end of the experiments using a bench top pH-meter (Edge, Hanna Instruments Inc., USA).

2.5. Phenolic characterization of the wines treated with grapevine-cane materials and cane extracts by HPLC-DAD-FLD

Low molecular weight phenolics were analyzed on an HPLC Agilent series 1200 equipped with a LiChrospher RP-18 5 μm, 250 × 4 mm column and a fluorescence and photodiode array detectors, according to the method proposed by Gómez-Alonso et al. (2007) [35]. Briefly, a ternary mobile phase gradient was used. Mobile phase A: $NH_4H_2PO_4$ 50 mM, pH 2.6. Mobile phase B: 20 % mobile phase A

Table 1
Spectrophotometric and enological parameters for the red wine (Cabernet Sauvignon) employed at the beginning of the assay (Time = 0 days).

Parameter	Value
pH	3.44 ± 0.010
TPC (mg GAE/L)	2201.3 ± 133.1
Tannins by MCP (mg CE/L)	1057.8 ± 43.2
Total sulfite (mg/L)	119.3 ± 3.1
Free sulfite (mg/L)	42.7 ± 2.1
Anthocyanins (mg/L)	313.3 ± 3.2
Catechins (mg/L)	411.0 ± 11.8
L-malic acid (g/L)	0.13 ± 0.01
Total acidity (g/L)	4.90 ± 0.23
Glucose-Fructose content (g/L)	15.40 ± 0.75
Potassium (mg/L)	809.5 ± 92.6
Calcium (mg/L)	74.5 ± 3.5
Copper (mg/L)	<0.4
Iron (mg/L)	3.81 ± 0.08
Glycerol (g/L)	5.34 ± 0.14
L*	7.43 ± 0.06
a*	36.44 ± 0.11
b*	12.79 ± 0.12
Color intensity	12.57 ± 0.10
Hue	0.743 ± 0.004

and 80 % acetonitrile. Mobile phase C: H_3PO_4 200 mM, pH 1.5. Flow rate 1 mL/min. Initial: 100 % A for 2 min, 92 % A and 8 % B at 5 min, 14 % B and 86 % C at 17 min, 18 % B and 82 % C at 22 min, 21 % B and 79 % C at 29.5 min, 33 % B and 67 % C at 55 min, 50 % B and 50 % C at 70 min for 5 min, 20 % B and 80 % C at 78 min for 3 min, 100 % A at 86 min. The DAD wavelengths of detection were 280 nm (monomeric flavanols), 320 nm (hydroxycinnamic acids), 360 nm (flavonols), and 520 nm (anthocyanins). Flavanols were also analyzed by fluorometric detection at $\lambda_{ex} = 280$ nm and $\lambda_{em} = 320$ nm. The identification of the phenolic compounds was carried out by comparing the absorption spectra and retention times with the standards available in the laboratory. Quantification was performed using external calibration curves. The concentration of phenolic compounds was expressed as mg/L.

2.6. Dissolved oxygen of the wines treated with grapevine canes

Dissolved oxygen was measured using a photoluminiscence-based oxygen detector and PSt3 oxygen sensors (NomaSense, Vinventions, USA), as described by Calderón et al. (2014) [36]. Sensors were glued at mid height inside the bottles (250 mL, glass) using food-grade silicone (NomaSense, Vinventions, USA), and to compensate for possible differences in the initial dissolved oxygen among bottles, the values were normalized at a 100 scale, according to Díaz et al. (2021) [37]. The oxygen readings started on the first day of the experiment (0 days) and were taken every 24 h until day 14. Given that no major variations in DO were observed thereafter, two final readings were then taken at days 19 and 24.

2.7. Solvent and reagents

Absolute ethanol, (*L*)-tartaric acid (99.5 %), sodium hydroxide (99 %), Folin-Ciocaletu reagent (2 N), gallic acid (98 %), sodium carbonate (99 %), ammonium sulphate (99 %), bovine serum albumin (99 %), potassium metabisulfite (97 %), potassium tartrate (99.5 %), hydrochloric acid (37 %), glacial acetic acid (100 %), sodium chloride (99.5 %) ammonium dihydrogen phosphate (99 %), orto-phosphoric acid (85 %), rutin hydrate (94 %), myricetin (96 %), quercetin hydrate (95 %), and malvidin 3-glucoside chloride (95 %) were purchased from Merck KGaA (Darmstadt, Germany). Methylcellulose (1500 cP viscosity), Trolox (97 %), *trans*-resveratrol (99 %), (+)-catechin hydrate (98 %), and epicatechin (98 %) were purchased from Sigma-Aldrich (St. Louis, MO, USA), whilst 2,2-difenil-1-picrilhidrazil (98 %), *trans*-ε-viniferin (90 %), petunidin-3-O-glucoside (98 %), and procyanidin B1/B2/B3 (98 %) were acquired from Cayman Chemical (Ann Arbor, Michigan, USA). HPLC-grade solvents, including acetonitrile, water, ethanol, and methanol were purchased from Scharlau (Barcelona, Spain).

2.8. Statistical analyzes

The statistical analyzes were performed using the software IBM® SPSS® Statistics 22.0. Descriptive statistics were employed to characterize the studied plant material, and the obtained wines, including the arithmetic mean and their standard deviation, according to the analyzed variables, and/or the treatments. One-way analysis of variance (ANOVA), and subsequently, Tukey multiple comparison *post hoc* test ($p < 0.05$) was applied to determine statistically significant differences among categorical variables (i.e., wood variety, wood format, and toast level). To obtain the spatial distribution and non-random patterns for the treated Cabernet Sauvignon red wines, a Principal Component Analysis (PCA) was performed, according to their chemical composition. Finally, to establish possible correlations between the different parameters in the red wines, a bivariate Pearson's correlation analysis was carried out (95 % confidence). The respective correlation coefficient (Pearson's *r*) was registered in each case, and their significance was denoted as * or ** according to their *p*-values ($p < 0.05$ and $p < 0.01$, respectively).

3. Results and discussion

3.1. Physico-chemical features of grapevine canes

3.1.1. Thermal stability

TGA is widely implemented for investigating and comparing thermal degradation events and kinetics, measuring the decrease in mass under controlled conditions, whilst temperature increases with time [26]. In this context, Fig. 1 shows the thermogram curve for

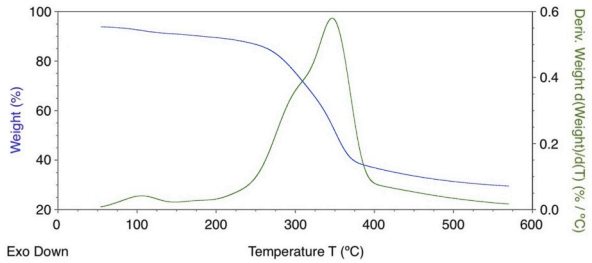

Fig. 1. Thermogravimetric analysis (blue) and first derivative of the thermogram (green) curves for Cabernet Sauvignon grapevine canes.

the Cabernet Sauvignon vine-canes, which correspond to the maximum degradation temperatures of their cell wall components. A mass loss close to 5 % can be seen between 50 and 100 °C, attributable to the moisture content and the presence of volatile compounds in the samples. In the interval between 100 and 180 °C, an additional mass loss of about 3 % was observed, corresponding to the constitutional water and the initial changes in lignin structure. The highest mass loss (more than 50 %) occurred in the range of 180 and 400 °C, in agreement with the results reported for vine canes from an Airén variety [18]. Finally, in the interval with temperatures higher than 400 °C, a final degradation step occurred, corresponding to the total decomposition of the wood constituents, including ash and combustion residues.

According to the TGA curves (Fig. 1), the highest mass loss was seen in the wide range between 180 and 400 °C, range that included the decomposition of lignin [15,18]. Consequently, based on these findings, it was established that temperatures of 180 °C or less may be suitable for the wood toasting process, without changing their structural composition.

3.1.2. Phenolic composition and antioxidant activity of grapevine cane extracts

To know the phenolic composition and the antioxidant activity of the Cabernet Sauvignon canes, an ultrasound assisted extraction was performed, which yielded 5 g of extract per 100 g of dry material, with a total phenolics content of 245.2 ± 29.3 mg GAE per g of extract (around 12500 mg GAE/Kg of dry material), as shown in Table 2. Similar studies conducted with grape wood from Turkey, Portugal, and Spain reported variable concentrations of phenolic compounds, ranging from 3000 to 36000 mg GAE/Kg of dry material, which depended not only on the grape variety, but also on the extraction methodology employed [4,13,38]. Like so, the antioxidant activity of the extracts was 30.2 ± 2.3 μg ext/mL (SC_{50}), which was lower than the results from Jesus et al. (2020) [39], who reported SC_{50} values between 760 and 1250 μg ext/mL. Again, varietal variations, and those related to extraction and analytical procedures may influence the antioxidant activity results.

According to the liquid chromatography analysis, the main phenolic observed was identified as *trans*-ε-viniferin (Fig. S2). This compound had the highest concentration (30.1 ± 0.2 mg per g of extract), around six times higher than *trans*-resveratrol, the second most concentrated compound in the extracts, with 5.3 ± 0.1 mg/g ext (Table 2). Contrary to these results, D'Eusanio et al. (2023) [40] reported that in pruning canes extracts from two cultivars of Lambrusco *Vitis vinifera* species the main compound was resveratrol, which was found to be around 4–6 times higher than *trans*-ε-viniferin. In that case, the authors combined ultrasound assisted extraction with maceration techniques, and controlled temperature [40]. Nevertheless, the resveratrol content in our samples was similar to that reported by Dorosh et al. (2020) [13], using ultrasound-assisted extraction, but higher than those of Çetin et al. (2011), in which maceration was used as the extractive method [4].

Trans-ε-viniferin and *trans*-resveratrol are stilbenoids that have been widely reported in grapevine shoots and canes [2,3,16,40–42]. Although *trans*-resveratrol has been extensively studied because its antioxidant capacity, or its potential anti-inflammatory, anticarcinogenic, and cardioprotective properties [43,44], the dehydrodimer *trans*-ε-viniferin, has been described as having stronger therapeutic and antioxidant properties than resveratrol, but potentially more toxic when highly concentrated [45].

Along these two main compounds, Table 2 also shows other phenolic compounds, including (+)-catechin (2.3 ± 0.3 mg/g ext) and (−)-epicatechin (1.4 ± 0.7 mg/g ext), whose concentrations were significantly lower than those reported in aqueous extracts of Airén vine-canes, which concentration was highly variable depending on the extraction method employed [16]. In addition, the extracts of the Cabernet Sauvignon vine canes also contained other minor compounds, such as procyanidin B1 and B3, which were quantified as procyanidin B1 equivalents, with a concentration of 3.4 ± 0.6 mg/g ext (around 150 mg/kg dry material). Procyanidin B2 was also detected in the extracts, but with a concentration bellow the lower limit of our calibration curve. In contrast, Cebrián-Tarancón et al. (2018) [46] reported higher values of procyanidins in toasted vine-canes from Airén and Cencibel cultivars, with values from 477 to 1079 mg of total procyanidins per Kg of dry material, but also using different extraction and measuring techniques. It has been demonstrated that extracts rich in dimeric procyanidins (B1, B2, B3), catechin, and epicatechin have significant antibacterial and antioxidant properties [47]. Therefore, vine cane extracts could have interesting applications due to their phenolic composition. However, the content and composition of phenolics in those extracts may vary largely depending on the extraction conditions (i.e., temperature, solid-to liquid ratio, extraction solvent, time, among others), the climate conditions in which the vines were grown, and the variety of canes employed [2,3,13,48,49].

Table 2
Phenolic composition of Cabernet Sauvignon grapevine cane extracts. Values are expressed as the mean ± standard deviation.

Parameters	Value
Yield of extraction (g ext/100 g dry wood)	5.0 ± 0.2
TPC (mg GAE/g ext)	245.2 ± 29.3
DPPH (SC_{50}) (μg ext/mL)	30.2 ± 2.3
(+)-catechin (mg/g ext)	2.3 ± 0.3
(−)-epicatechin (mg/g ext)	1.4 ± 0.7
Procyanidin B1/B3 (mg/g ext)[a]	3.4 ± 0.6
Procyanidin B2 (mg/g ext)	<1
Trans-resveratrol (mg/g ext)	5.3 ± 0.1
Trans-ε-viniferin (mg/g ext)	30.1 ± 0.2

[a] Expressed as procyanidin B1 equivalents.

3.2. Maceration of grapevine canes in wine-like model solution

Considering the importance of investigating the transference of phenolic compounds from vine canes to wines, before using them as an enological additive [21], an initial experiment was conducted, using wine-like model solution. Fig. 2 and Table S1 show that the wine-like model solutions reached a maximum content of phenolics compounds at 24 days of maceration in all treatments, including the model solutions treated with oak wood (control). Nevertheless, the model solutions obtained using untoasted powder vine canes showed a different behavior, reaching the maximum level at 12 days of maceration, which rapidly decayed at 24, and 36 days. In comparison, other studies using vine canes from Airén and Cencibel cultivars, showed an important transference of enological compounds at 35 days of maceration, using the same dose (12 g/L) [21]. On the contrary, it has been reported that longer maceration times are required when using oak wood, but that lower doses may suffice [28,50]. In this work, a dose of 12 g/L of oak chips was also used as a positive control, reaching significantly higher phenolic content than the treatments using vine canes at 36 days of maceration (Table 3; Fig. S3).

On the other hand, the model solutions treated with low, and high toasting level vine canes showed similar phenolic contents (71.27–77.27 mg GAE/L), but significantly higher than the samples treated with untoasted vine canes (55.43–60.10 mg GAE/L), with the exception of the samples treated with untoasted powder wood (76.43 mg GAE/L) (Table 3). Instead, the antioxidant activity of the model solutions showed slight differences among treatments (37.09–46.30 mg TE/L), but similar to the model solutions treated with oak wood (45.81 $\pm$ 0.61 mg TE/L) (Table 3). As a reference for comparison, a total value of phenolics compounds between 35 and 57 mg/L was reported for model solutions treated with vine canes from two cultivars in Spain using liquid chromatography as quantitative method [21].

3.3. Maceration of grapevine canes in red wines

Given the results of section 3.2, an experiment with Cabernet Sauvignon wine macerated with toasted Cabernet Sauvignon canes was also performed. Untoasted grapevine canes were not used in this experiment, due to their lower contribution to the phenolic content of wine-like model solutions and its rapid decrease over time (Fig. 2, Table 3). Consequently, six treatments were evaluated, combining vine cane format and toasting level (i.e., low toasting level (LTL)-Powder, LTL-Granules, LTL-Discs and high toast level (HTL)-powder, HTL-Granules, HTL-Discs), and two control treatments (i.e., untreated control wine and oak control). The Cabernet Sauvignon wine was characterized according to its spectrophotometric and enological parameters at the beginning of the assay, as shown in Table 1.

The total phenolics content (TPC) of the wines treated with powder grapevine canes at a low toasting level, was significantly lower than the controls (Table 4), and also lower than the wines treated with granules and discs. Like so, the antioxidant activity of the wines treated with grapevine canes was lower than the control wine, regardless of the toasting level. Total phenolic measurements with the Folin-Ciocalteu reagents have been shown to be influenced by substances in solution other than phenolics, including sulfites, sugars, and proteins, possibly raising the TPC [51]. Moreover, the powder format of grapevine canes may be promoting the removal of phenolics or other constituents that are reactive towards the Folin-Ciocalteu reagent. This observation is in line with previous reports showing that insoluble plant fibers could potentially be used for the fining of wine phenolics [52].

Tannins, anthocyanins, and polymeric pigments are essential phenolic constituents of red wine because they provide color, color

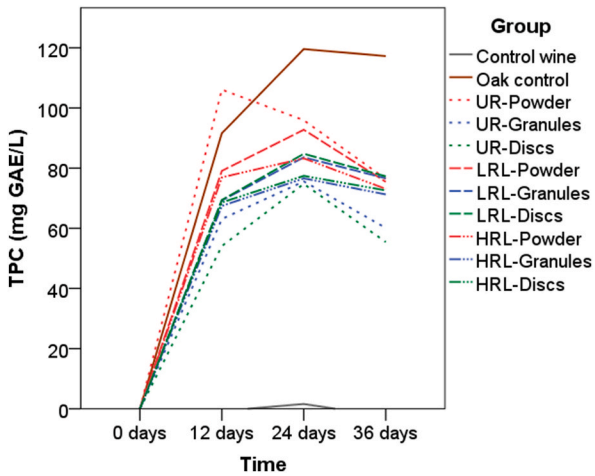

Fig. 2. Total phenolics content at 0, 12, 24, and 36 days in wine-like model solutions treated with vine-cane wood at different toasting levels (UR: Untoasted wood, LTL: Low toast level, HTL: High toast level), and different wood formats.

Table 3
Total phenolics content (TPC) and antioxidant activity (AOx) of model wines with 36 days of grapevine canes wood maceration. Letters (a-c): indicate significant differences between treatments according to the Tukey's test ($\alpha = 0.05$).

Treatment	TPC (mg GAE/L)	AOx (mg TE/L)
Woodless control	0.00 ± 0.00 a	0.86 ± 0.53 a
Oak control	117.27 ± 7.52 d	45.81 ± 0.61 d
Untoasted Wood		
Powder	76.43 ± 2.73 c	46.30 ± 4.98 d
Granules	60.10 ± 3.52 b	38.68 ± 2.52 bc
Discs	55.43 ± 7.17 b	38.70 ± 3.37 bc
Low toast level		
Powder	75.43 ± 3.27 c	39.74 ± 1.14 bc
Granules	76.60 ± 2.07 c	38.42 ± 0.62 bc
Discs	77.27 ± 2.79 c	41.56 ± 1.13 c
High toast level		
Powder	73.10 ± 2.53 c	39.31 ± 0.80 bc
Granules	71.27 ± 3.25 c	37.09 ± 0.71 b
Discs	72.60 ± 2.59 c	39.21 ± 1.02 bc

stability, and mouthfeel properties like astringency [53]. Our results show that the wines treated with granules and discs, at a high toasting level, have a condensed tannin concentration near 1050 mg/L (catechin equivalents), which was equivalent than that of the control wines (i.e., 1021.7 ± 34.1 and 1085.9 ± 16.6 mg/L, catechin equivalents). Alternatively, the powder format at high toasting level reduced the concentration of condensed tannins, in comparison with the untreated control wine, but this was not seen in the treatments in which a low toasting level was employed. In addition, most of the treatments, with the exception of granules and discs at high toasting level, caused a reduction of total catechins. These observations suggest that the powder vine canes may not be adequate for their use as an enological additive, unless a reduction in the concentration of wine phenolics is desired, and that the dynamics of phenolic release and adsorption into the biomass employed for maceration should be carefully studied.

On the other hand, the content of large polymeric pigments (LPP) was significantly higher in both the untreated and oak control samples (0.298 and 0.286 AU, respectively) compared to the vine-canes treated samples. Instead, slight differences were found in small polymeric pigments (SPP), but the treatment showing the lowest SPP value was the low toast vine-cane powder.

Regarding the individual CIELab color parameters, no significant differences were observed in the L*, a*, and b* among all treatments, probably because of the high levels of phenolic compounds of the wine employed, as previously argued elsewhere [25]. Lower L* (lightness) numbers are indicative of darker tones leaning towards black, positive a* (green to red) values represent red tones, whilst positive b* results are indicative of yellow tones. Instead, the color intensity of the treated samples was slightly lower than the controls. Nevertheless, the wines treated with low toast level grapevine granules did not show differences with the control. With regards to hue (tonality), all wines treated with toasted vine canes had higher values that both control treatments. Hue values represent the ratio of 420:520 nm light absorption, and larger values could be indicative of more yellow color in the samples due to the release of yellow-absorbing compounds from the cane materials. The prior was observed in the hue analyses, but no so in the case of the CIELab measurements. Like so, the ΔE^*_{ab} parameter, showed values lower than 2 units in all treatments when compared to the control wine, but between 2 and 3 for almost all vine-cane treated wines when compared with the oak control wine (Table 4). Although, the ΔE^*_{ab} values could be important for the wine industry, it is not possible to establish a concrete value for color discrimination because many factors condition this limit. Regardless of the prior, Fanzone et al. (2021) [25] suggest that there may be a relative visual discriminating threshold in wines for values between 3 and 5 units.

On the other hand, most of the quantified low molecular weight phenolics decreased their concentration when vine canes or oak wood was applied to the wines. Regarding anthocyanins, the most noticeable changes were observed for petunidin 3-*O*-glucoside and malvidin 3-glucoside which concentration was lower in the vine-cane treated samples. Like so, a significant reduction in the concentration of total anthocyanins was observed in relation to the control wine (Table 4, Fig. S4). This reduction was more apparent in wines treated with powder vine canes, both in high and low toast level (with a reduction of around 23 % and 20 % respectively). On the other hand, some flavonols, including rutin, myricetin, and quercetin also decreased their concentration in relation to the control wine, but in a global comparison of the phenolic composition, the results were similar to the oak control wine.

Despite the decrease in total anthocyanins, probably, due to precipitation or adsorption onto the biomass employed, no significant alterations in the L* a* b* parameters were observed. It is well known that red wine color may not be solely explained by the content of free anthocyanins, but that other phenomenon such as co-pigmentation, the presence of polymeric pigments, and sulfite bleaching should also be considered [53,54].

With regards to wine pH, it was observed that the wines treated with vine canes had a slightly higher pH than the control wines

Table 4
Chemical composition of the resulting Cabernet Sauvignon red wines at 24 days of maceration with toasted vine canes, according to each treatment, including the spectrophotometric data, CIELab color parameters, enological parameters, and their phenolic composition (low molecular weight phenolic components). Different letters (a-f) indicate significant differences according to the Tukey's test (α = 0.05).

Parameters	Controls		Low toast level			High toast level		
	Control wine	Oak control	Powder	Granules	Discs	Powder	Granules	Discs
Spectrophotometric parameters								
TPC (GAE, mg/L)	2098.0 ± 40.0 b	2111.3 ± 69.3 b	1691.3 ± 111.8 a	2006.3 ± 78.5 b	1944.7 ± 145.1 b	1908.0 ± 87.9 ab	1966.3 ± 50.1 b	2034.7 ± 46.5 b
AC (TE, mg/L)	1850.2 ± 41.4 c	1774.0 ± 11.5 bc	1575.1 ± 62.4 a	1648.7 ± 50.7 ab	1690.9 ± 52.4 ab	1631.8 ± 100.5 ab	1594.5 ± 42.6 a	1597.9 ± 19.8 a
Tannins (CE, mg/L)	1021.7 ± 34.1 bcd	1085.9 ± 16.6 c	998.1 ± 63.0 bcd	959.1 ± 40.2 ab	928.8 ± 28.6 ab	894.4 ± 19.2 a	1052.0 ± 13.0 cd	1046.8 ± 45.9 cd
LPP (AU)	0.298 ± 0.007 f	0.286 ± 0.011 f	0.229 ± 0.001 c	0.243 ± 0.001 cd	0.263 ± 0.003 e	0.174 ± 0.002 a	0.252 ± 0.004 de	0.211 ± 0.004 b
SPP (AU)	0.461 ± 0.003 bcd	0.470 ± 0.007 cd	0.425 ± 0.006 a	0.476 ± 0.003 d	0.451 ± 0.002 b	0.453 ± 0.002 bc	0.474 ± 0.013 d	0.501 ± 0.002 e
Color parameters								
Color intensity	12.75 ± 0.26 bc	13.60 ± 1.15 c	11.14 ± 0.44 a	12.17 ± 0.63 abc	11.64 ± 0.37 ab	11.26 ± 0.08 a	11.69 ± 0.19 ab	11.80 ± 0.26 ab
L*	7.10 ± 0.61 a	6.53 ± 0.91 a	7.67 ± 0.15 a	7.17 ± 0.40 a	7.83 ± 0.23 a	7.47 ± 0.38 a	7.83 ± 0.25 a	7.43 ± 0.40 a
a*	35.77 ± 1.17 a	34.61 ± 2.02 a	36.14 ± 0.44 a	35.65 ± 0.67 a	36.74 ± 0.13 a	35.91 ± 0.62 a	36.68 ± 0.24 a	36.03 ± 0.66 a
b*	12.18 ± 1.03 a	11.25 ± 1.54 a	13.14 ± 0.31 a	12.33 ± 0.73 a	13.45 ± 0.34 a	12.85 ± 0.65 a	13.47 ± 0.37 a	12.75 0.70 a
Tonality	0.741 ± 0.005 a	0.766 ± 0.002 b	0.836 ± 0.012 de	0.819 ± 0.018 cd	0.812 ± 0.007 cd	0.853 ± 0.003 e	0.819 ± 0.006 cd	0.809 ± 0.008 c
ΔE^*_{ab} (Control wine)	–	1.41	1.41	0.00	1.73	1.41	1.73	1.00
ΔE^*_{ab} (Oak control)	1.41	–	2.45	1.41	3.00	2.45	3.0	2.24
Enological parameters								
Total sulfite (mg/L)	104.7 ± 1.5 b	93.3 ± 2.5 a	90.0 ± 3.5 a	95.0 ± 5.6 a	95.0 ± 3.0 a	89.0 ± 1.0 a	95.0 ± 1.7 a	95.0 ± 4.6 a
Free sulfite (mg/L)	37.3 ± 1.2 c	26.7 ± 0.6 ab	25.3 ± 1.5 a	28.3 ± 3.1 ab	30.3 ± 2.1 b	26.0 ± 1.0 ab	29.0 ± 1.7 ab	30.7 ± 1.5 b
Anthocyanins (mg/L)	294.7 ± 5.7 e	276.4 ± 7.6 d	237.3 ± 4.6 a	259.7 ± 6.1 b	261.3 ± 5.7 bc	228.9 ± 1.9 a	266.5 ± 2.9 bcd	274.8 ± 4.3 cd
Total catechins (mg/L)	388.9 ± 10.2 e	380.5 ± 0.5 de	352.0 ± 3.7 ab	364.0 ± 8.7 bc	370.6 ± 2.0 cd	345.0 ± 3.7 a	377.9 ± 3.8 cde	392.9 ± 5.6 e
L-malic acid (g/L)	0.103 ± 0.026 a	0.099 ± 0.018 a	0.116 ± 0.026 a	0.125 ± 0.008 a	0.112 ± 0.012 a	0.127 ± 0.008 a	0.127 ± 0.012 a	0.127 ± 0.007 a
pH	3.50 ± 0.006 a	3.51 ± 0.010 a	3.59 ± 0.006 b	3.57 ± 0.012 b	3.57 ± 0.012 b	3.59 ± 0.006 b	3.58 ± 0.001 b	3.58 ± 0.006 b
Low molecular weight phenolic compounds								
Flavanols (mg/L)								
(+)-Catechin	11.84 ± 1.27 b	9.41 ± 0.63 a	10.16 ± 0.39 ab	9.56 ± 0.52 a	10.16 ± 0.21 ab	8.56 ± 0.44 a	8.71 ± 0.41 a	9.54 ± 0.98 a
(−)-Epicatechin	8.18 ± 0.86 b	6.45 ± 0.45 a	6.25 ± 0.22 a	6.41 ± 0.24 a	6.53 ± 0.24 a	5.49 ± 0.10 a	5.90 ± 0.20 a	6.20 ± 0.42 a
Procyanidin B1/B3[a]	18.35 ± 1.73 c	15.34 ± 0.87 b	14.04 ± 0.48 ab	14.53 ± 0.68 ab	14.69 ± 0.59 ab	12.58 ± 0.24 a	13.59 ± 0.26 ab	14.17 ± 0.79 ab
Procyanidin B2	16.74 ± 1.19 b	14.74 ± 1.11 ab	13.28 ± 0.66 a	13.84 ± 0.63 a	13.96 ± 0.56 a	12.43 ± 0.60 a	13.14 ± 0.71 a	13.59 ± 1.09 a
Σ Flavanols	55.12 ± 4.81 b	45.95 ± 3.06 a	43.72 ± 1.58 a	44.33 ± 1.59 a	45.35 ± 1.21 a	39.06 ± 1.01 a	41.35 ± 1.13 a	43.51 ± 3.13 a
Phenolic acids (mg/L)								
Gallic acid	40.62 ± 0.67 bc	39.85 ± 0.31 ab	41.17 ± 0.40 c	39.43 ± 0.21 a	39.71 ± 0.18 ab	41.59 ± 0.15 c	39.46 ± 0.23 a	39.81 ± 0.67 ab
Caffeic acid	5.32 ± 0.09 b	4.83 ± 0.08 a	4.81 ± 0.09 a	4.74 ± 0.13 a	4.77 ± 0.11 a	4.76 ± 0.13 a	4.67 ± 0.13 a	4.79 ± 0.21 a
N.i.[b]	14.51 ± 0.25 a	14.38 ± 0.30 a	14.34 ± 0.50 a	13.82 ± 0.23 a	13.54 ± 0.08 a	14.52 ± 0.47 a	13.86 ± 0.50 a	14.09 ± 0.70 a
Σ Phenolics acids	60.45 ± 0.68 bc	59.06 ± 0.16 abc	60.32 ± 0.23 bc	58.00 ± 0.10 a	58.02 ± 0.22 a	60.87 ± 0.66 c	57.99 ± 0.59 a	58.69 ± 1.45 ab
Anthocyanins (mg/L)								
Petunidin 3-*O*-glucoside	18.88 ± 0.22 e	15.63 ± 0.58 d	12.94 ± 0.32 b	14.57 ± 0.53 cd	14.53 ± 0.42 cd	11.71 ± 0.42 a	14.27 ± 0.43 c	14.58 ± 0.46 cd
Malvidin 3-glucoside	69.66 ± 1.36 d	57.19 ± 2.33 c	47.85 ± 1.29 ab	53.73 ± 1.77 c	53.65 ± 1.62 c	43.91 ± 2.13 a	52.62 ± 1.99 bc	53.65 ± 1.91 c
Σ Anthocyanins[c]	88.54 ± 1.55 d	72.83 ± 2.90 c	60.79 ± 1.61 ab	68.30 ± 2.27 c	68.18 ± 1.98 c	55.61 ± 2.54 a	66.89 ± 2.40 bc	68.21 ± 2.34 c
Flavonols (mg/L)								
Rutin	34.91 ± 1.18 e	32.00 ± 0.50 d	23.62 ± 0.33 ab	25.52 ± 0.80 bc	25.56 ± 0.76 bc	22.41 ± 0.15 a	24.64 ± 0.44 abc	26.55 ± 1.38 c
Myricetin	7.55 ± 0.39 f	5.48 ± 0.18 e	4.17 ± 0.12 ab	4.69 ± 0.08 bcd	4.94 ± 0.10 cde	3.91 ± 0.04 a	4.44 ± 0.10 abc	4.99 ± 0.27 de
Quercetin	13.69 ± 0.14 e	7.16 ± 0.50 d	4.57 ± 0.21 a	5.81 ± 0.27 bc	6.46 ± 0.30 cd	4.37 ± 0.26 a	5.07 ± 0.34 ab	6.78 ± 0.35 d
Σ Flavonols	56.15 ± 1.17 f	44.64 ± 0.50 e	32.37 ± 0.51 ab	36.02 ± 0.92 cd	36.96 ± 1.01 d	30.69 ± 0.43 a	34.15 ± 0.40 bc	38.32 ± 1.92 d

[a] Expressed as procyanidin B1 equivalents.
[b] Not identified acid, expressed as caffeic acid equivalents.
[c] Include all signals quantified at 520 nm. Expressed as malvidin 3-glucoside equivalents.

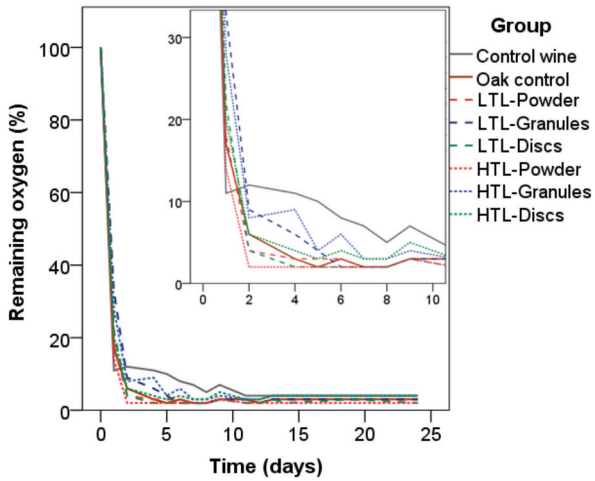

Fig. 3. Oxygen consumption in the obtained Cabernet Sauvignon red wines macerated with grapevine-cane wood in their different formats. LTL: Low toast level. HTL: High toast level.

(3.58 vs. 3.50). It has been reported that vine shoots from different cultivars contain a relevant amount of minerals, mainly, potassium and calcium [4,16,55], which potentially could be released to the wine, causing a pH rise. As reported elsewhere, these cations may react with tartaric acid, forming insoluble salts and therefore increasing the wine's pH [56,57]. On the other hand, it has been reported that variations of pH may impact the color, sensorial properties, and the phenolic composition of red wines [58–61]. However, these events are a result of more complex processes, which cannot be explained solely by the changes on pH [61].

Contrarily to previous studies in which vine cane wood was applied to red wines [18,20,23,24,62], and although the presence of stilbenoids in the vine canes employed (Table 2), *trans*-resveratrol or *trans*-ε-viniferin were not detected in quantifiable amounts in the treated wines. This could be explained by the fact that the stilbenes content decreases with increasing toasting temperature, probably, because of their thermolability, regardless of the grape cultivar or sampling time [40], or that longer maceration times would have been required to extract these substances.

An additional aspect to take into account, is that the dissolved oxygen of the wines treated with vine canes or oak were lower than the untreated control (Fig. 3). This is in line with previous reports indicating that the release of phenolic compounds from oak-wood treatments to wines may contribute to lowering the dissolve oxygen levels, as a result of oxidative reactions between those phenolics and the oxygen in sollution [63–65]. Variables such as wood source, toasting level and wood format are relevant in studies involving oxygen consumption and wine aging [63–66]. For instance, the oxygen trapped in the oak wood porosity may slowly and continuously diffuse into the wine, encouraging the reactions usually related with wine aging [67,68]. Our results show the wines treated with vine cane discs and granules exhibited a similar behavior to the oak control, both at low and high toasting levels. The oxygen values (at the end of the experiment) showed significant correlations with most of the phenolic compounds, including total anthocyanins (Pearson's $r = 0.748$, $p = 3 \times 10^{-5}$) and catechins (Pearson's $r = 0.783$, $p = 6 \times 10^{-6}$) (Table 5), which is in agreement with previous reports [64,65]. Significant correlations with other parameters were also observed (Tables 5 and 6), including total and free sulfites (Pearson's $r =$ 0.654 and 0.714, $p < 0.001$, respectively), but correlations with color parameters, which could be associated to oxidative changes [37], were not observed.

It is important to note that many studies on the addition of wood during wine aging have used micro-oxygenation [64–66], and most of that experiments were conducted in tanks with longer maceration times. Therefore, as a future projection, it would be interesting to study the oxygen consumption in wines macerated with Cabernet Sauvignon vine canes under industrial conditions.

Finally, the Principal Component Analysis performed (Fig. 4a) showed that the main 3 principal components explained 78 % of the variance in the dataset. Red wines treated with different formats of grapevine-canes residues showed clear differences in their spatial distribution when compared to the untreated control wines, possibly explained by their content of phenolics (e.g., catechin), sulfites, pH and other less contributing variables (Fig. 4b). The oak control wines clearly differ from the wines treated with grapevine-canes in powder format, most likely driven by their differences in total and polymerized phenolics (e.g. tannins) as observed in Fig. 4b and Table 4. Instead, the wines treated with granules and discs formats grouped between the oakwood and grapevine-powder treated wines. The prior agrees with other investigations in which similar vine shoot formats are recommended as enological additives [18,24,62].

Table 5
Correlation matrix (Pearson's *r* coefficients) between pH, oxygen, and spectrophotometric and enological parameters in wines treated with grapevine canes. Significant correlations are indicated with * ($p < 0.05$) and ** ($p < 0.01$).

	pH	TPC	Aox	Tannins	LPP	SPP	Total sulfite	Free sulfite	Antho-cyanins	Catechins	L*	a*	b*	Int.	Ton.
pH	1														
TPC	−0.572**	1													
Aox	−0.831**	0.563**	1												
Tannins	−0.451*	0.308	0.234	1											
LPP	−0.810**	0.441*	0.674**	0.475*	1										
SPP	−0.076	0.641**	0.004	0.357	0.015	1									
Total sulfite	−0.547**	0.466*	0.627**	0.281	0.621**	0.250	1								
Free sulfite	−0.484*	0.487*	0.606**	0.182	0.515*	0.286	0.931**	1							
Anthocyanins	−0.724**	0.665**	0.624**	0.605**	0.767**	0.530**	0.819**	0.776**	1						
Catechins	−0.538**	0.645**	0.399	0.669**	0.570**	0.651**	0.665**	0.680**	0.911**	1					
L*	0.573**	−0.359	−0.492*	−0.362	−0.263	−0.152	−0.107	−0.071	−0.283	−0.236	1				
a*	0.417*	−0.210	−0.359	−0.313	−0.119	−0.044	0.020	0.058	−0.116	−0.104	0.965**	1			
b*	0.576**	−0.360	−0.495*	−0.372	−0.269	−0.145	−0.112	−0.077	−0.288	−0.244	0.999**	0.968**	1		
Int.	−0.798**	0.565**	0.626**	0.491*	0.605**	0.275	0.325	0.272	0.601**	0.516**	−0.828**	−0.721**	−0.825**	1	
Ton.	0.922**	−0.630**	−0.797**	−0.498*	−0.838**	−0.245	−0.738**	−0.699**	−0.889**	−0.740**	0.436*	0.266	0.439*	−0.741**	1
Oxygen	−0.338	0.378	0.214	0.552**	0.389	0.491*	0.654**	0.714**	0.748**	0.783**	0.013	0.104	0.003	0.195	0.729**

Table 6
Correlation matrix (Pearson's *r* coefficients) between pH, oxygen, and phenolic compounds in wines treated with grapevine canes. Significant correlations are indicated with * ($p < 0.05$) and ** ($p < 0.01$).

	pH	Catechin	Pro- B1/B3	Epicatechin	Pro- B2	Pet-3-O-Glu	Mal-3-Glu	Rutin	Myricetin	Quercetin	Caffeic acid	Gallic acid	Oxygen
pH	1												
Catechin	−0.487*	1											
Procyanidin B1/B3	−0.748**	0.853**	1										
Epicatechin	−0.660**	0.884**	0.985**	1									
Procyanidin B2	−0.732**	0.801**	0.952**	0.929**	1								
Petunidin-3-O-Glu	−0.826**	0.696**	0.907**	0.867**	0.853**	1							
Malvidin-3-Glu	−0.819**	0.710**	0.907**	0.870**	0.838**	0.996**	1						
Rutin	−0.945**	0.586**	0.843**	0.765**	0.847**	0.914**	0.896**	1					
Myricetin	−0.844**	0.684**	0.876**	0.850**	0.839**	0.949**	0.946**	0.927**	1				
Quercetin	−0.792**	0.739**	0.893**	0.877**	0.842**	0.936**	0.943**	0.885**	0.982**	1			
Caffeic acid	−0.638**	0.657**	0.758**	0.769**	0.802**	0.681**	0.659**	0.750**	0.807**	0.816**	1		
Gallic acid	0.127	0.180	0.030	0.079	0.016	−0.268	−0.243	−0.144	−0.096	−0.012	0.266	1	
Oxygen	−0.338	0.380	0.506*	0.506*	0.462*	0.670**	0.666**	0.511*	0.622**	0.636**	0.457*	−0.277	1

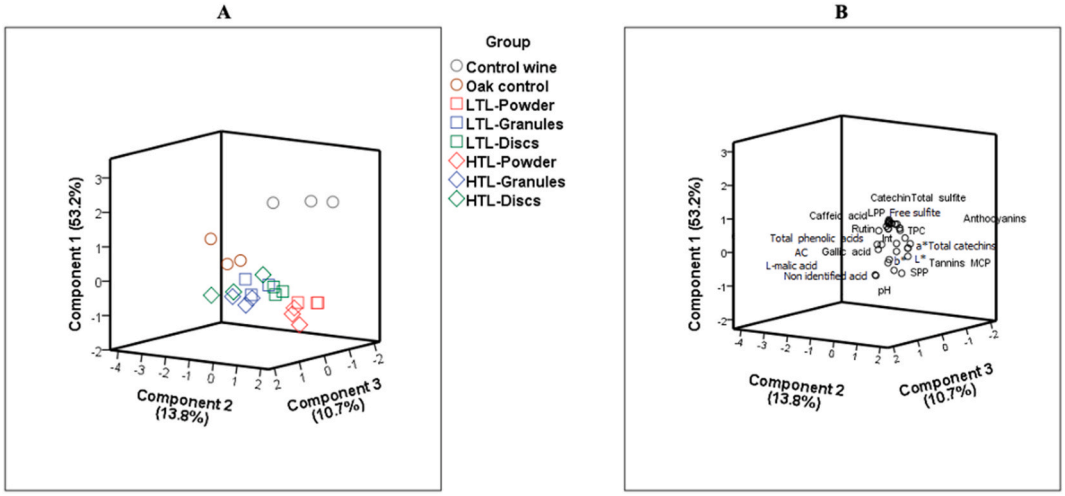

Fig. 4. Three-dimensional principal component analysis (A) and loading plot (A) of the physico-chemical variables analyzed in Cabernet Sauvignon wine treated with grapevine-cane chops compared to untreated and oak-treated controls.

4. Conclusions

The Cabernet Sauvignon vine canes tested have an important content of phenolic compounds, mainly, *trans*-ε-viniferin and *trans*-resveratrol, and their extracts showed a significant antioxidant activity. However, from the maceration trials performed in red wine it was noticed that the powder format of grapevine canes caused significant decreases in total phenolics, condensed tannins, antioxidant activity, total anthocyanins, and catechins. Instead, granules and discs produced no major variation in phenolics when compared to the controls, suggesting that these types of wood format should be preferred, and that longer maceration times may be required to have significant increases in the concentration of wine phenolics.

CRediT authorship contribution statement

Verónica R. Olate-Olave: Writing – review & editing, Writing – original draft, Visualization, Supervision, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Liudis L. Pino-Ramos:** Writing – review & editing, Investigation, Formal analysis. **Paula A. Peña-Martínez:** Writing – review & editing, Investigation, Formal analysis. **Ricardo I. Castro:** Writing – review & editing, Investigation, Formal analysis. **Marcelo Muñoz-Vera:** Writing – review & editing, Investigation, Formal analysis. **Sergio Reyes-Manríquez:** Writing – review & editing, Investigation, Formal analysis. **Gerard Casaubon:** Writing – review & editing, Resources, Funding acquisition. **V. Felipe Laurie:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization.

Data availability statement

Not applicable.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used Grammarly in order to review spelling and grammar of the manuscript. After using this tool, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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