Research Article

Chemical exchange in the vine shoots–wine system when used as an innovative enological procedure

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Abstract

BACKGROUND: Pruned vine shoots prepared as toasted fragments (SEGs) have recently been proposed as enological additives capable of producing differentiated quality wines. In this work, the composition of phenolic and volatile compounds of SEGs, before and after contact with wines, has been studied.

RESULTS: SEGs from Tempranillo and Cabernet Sauvignon were used, which were kept in contact for 30 days with red wines made with the same varieties. Phenolic compounds were the ones with the highest sorption in SEGs, but a variety-dependent behavior was observed in anthocyanins and flavonols, with an increase in some malvidin derivatives only in Tempranillo wine and an increase in (−)-epicatechin in SEGs and Cabernet Sauvignon wine. trans-Resveratrol was transferred from SEGs to wine but also increased in SEGs regardless of the variety used. The volatile compounds that were most retained in SEGs were phenylethyl alcohol and ethyl lactate, but in lower proportions than the phenolic compounds and without important changes in wines.

CONCLUSION: The high content of phenolic compounds in SEGs after their use as enological additives suggests that they could be considered as a source of anthocyanins and as raw materials for phenolic compounds with recognized antioxidant properties.
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Supporting information may be found in the online version of this article.

Keywords: Cabernet Sauvignon; phenolics; SEGs–wine balance; sorption; Tempranillo; volatiles

INTRODUCTION

Toasted pruned vine shoots represent a promising new enological tool for developing wines with a chemical and organoleptic differentiated profile,[1,2] allowing the resources of the vineyard to be returned to the wine through a 'circular process'.

The phenolic, volatile, and mineral composition of vine-shoots has been widely studied.[3-8] Along with the previous characterization, the evolution of fungicide residues in vine-shoots after pruning and toasting treatment[2] and their possible toxicity in terms of acute toxicity and cytotoxicity[9] have also been studied, suggesting that wines macerated with their own vine-shoots are safe for consumption. Nevertheless, similar to other enological tools, the use of vine shoots as an enological additive is a complex process dependent not only on the chemical composition but also on the interaction of various factors, such as the effect of fragment size, toasting treatment, dosage, contact length, and time of addition.[5,10,11]

Vine shoots toasted fragments prepared as enological additives, called SEGs (a term derived from 'shoot from vines–enological–granule'), contribute a high complexity to wine due to an exchange between the chemical compounds of wood and wine.

In fact, the effect of using vine shoots as an enological additive on the chemical composition of wines has been studied in Airén white wines and in Tempranillo, Malbec, and Bonarda red wines,[1,11,12] the results of which have proved a greater differentiation of the compounds involved in the sensory profile of wines.

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This differentiation may be a consequence of both the transfer of phenolic and volatile compounds by the vine shoots to the wine[1,11] and the absorption of some of these compounds by the vine shoots from wine, which has recently been proposed by Cebrián-Tarancón *et al.*[12] These authors observed a decrease in the anthocyanin and other compounds of wine that they attributed to vine shoots absorption, which increased with contact time and a higher dose of vine shoots. These results are in agreement with previous studies for oak wood,[13] where the sorption was clearly dependent on the surface of wood in contact with the wine or, in terms of volatile compounds, the influence of particle size on the wood–wine sorption equilibrium.[14]

However, to the best of our knowledge, the effect of vine shoots contact with wine on the chemical composition of the final product has not been carried. However, this is an extremely important factor to understand the modifications that take place during vine shoots–wine matrix contact. Moreover, considering that wood could be enriched with different compounds present in the wine, detailed knowledge of vine shoots composition after contact is equally important, especially to propose subsequent reuses.

Therefore, the goal of this study was to identify the exchanges in terms of phenolic and volatile fractions in Tempranillo and Cabernet Sauvignon wines made with vine shoots as well as in the vine shoots' composition after contact with wine.

MATERIALS AND METHODS

Plant material
Vine shoots were pruned in January 2020 from Tempranillo (VIVC: 12350) and Cabernet Sauvignon (VIVC: 1929) red *Vitis vinifera* L. cultivars grown in the Pago de La Jaraba winery (Castilla-La Mancha, Spain). The grapevines were planted as a vertical shoot position trellis, pruned to bilateral cordon, and grown in an ecological system under non-irrigation conditions. After pruning, the samples were stored intact in the dark at room temperature (18 ± 3 °C) for 6 months and then ground into granules ranging from 2 mm to 2 cm using a hammer miller (Skid Sinte 1000; LARUS Impianti, Zamora, Spain). Then, the samples were subjected to a toasting process in an oven with air circulation (Heraeus T6; Heraeus, Hanau, Germany) at 180 °C for 45 min, according to Cebrián-Tarancón *et al.*[5]

Winemaking with SEGs
Grapes of both varieties were harvested from the same vineyards where the vine shoots were pruned and grape enological parameters were analyzed according to the International Organisation of Vine and Wine (OIV) methods[15] (Supporting Information Table S1). Musts were inoculated simultaneously with the commercial *Saccharomyces cerevisiae* strain Uvaferm HPS active dry yeast (Lallemand, St Simon, France) to perform the alcoholic fermentation and the commercial *Oenococcus oeni* strain Lalvin VP41 (Lallemand, St Simon, France) to develop the malolactic fermentation. Vinifications were performed in duplicate at a controlled temperature (20 ± 2 °C), obtaining around 200 L of wine for each replicate by using 'always full' 500 L stainless steel tanks. Finally, potassium metabisulfite was added after malolactic fermentation to give a total sulfur dioxide concentration of 50 mg L^{-1}. Alcoholic fermentation, which lasted for 10 days, was followed by a daily determination of the must temperature and density. Malic acid was measured every 2 days, and when its concentration was below 0.1 g L^{-1} the malolactic fermentation was considered complete. SEGs were added to wines at a dose of 24 g L^{-1} after malolactic fermentation and were removed after 30 days of maceration, according to Cebrián-Tarancón *et al.*[12] The mean values (plus/minus standard deviation) of the enological parameters of wine, analyzed according to OIV methods,[15] are summarized in Supporting Information Table S2.

Vine shoot extraction procedures
Extraction of compounds from SEGs before and after contact with wine was studied with the aim of understanding the release/sorption of volatile and phenolic compounds by wood during the contact. For that, two aliquots (20 g each one) of SEGs were taken randomly before being added to the tank and another two aliquots (20 g each one) after contact.

For the analysis of SEGs, each aliquot of 20 g was moisturized with 100 g of an ethanol–water solution (12.5%, pH 3.62) during 8 h at room temperature. After that, another 1792 x g of the same solution was added, and the extraction was carried out using a NEOS microwave device (Milestone Srl, Sorisole, Italy) according to the method of Cebrián *et al.*[3] Briefly, the extraction conditions were set at 75 °C (600 W) for 12 min with reflux to prevent dryness. All extracts were centrifuged at 4000 rpm for 10 min, and the supernatant was then separated. Subsequently, the solid sample was extracted another two times until exhaustion using the same quantity of ethanolic solution (100 g). The three extracts were mixed and kept at 5–7 °C till their analysis. All extractions were carried out in duplicate for each variety and sampling time.

Determination of volatile compounds by gas chromatography–mass spectrometry
Wine volatiles were determined according to the methodology of Sánchez-Gómez *et al.*[16] Wine volatiles were extracted by means of stir-bar sorptive extraction (polydimethylsiloxane; 10 mm length; 0.5 mm film thickness) by stirring 25 mL at 500 rpm for 60 min. For SEGs extracts, 22 mL was stirred at 500 rpm for 60 min at 60 °C and volatiles were isolated by means of headspace stir-bar sorptive extraction according to the Sánchez-Gómez *et al.*[6] method, slightly modified. Later, for both wines and SEGs extracts, analysis was performed using an automated thermal desorption unit (Gerstel, Mülheim and der Ruhr, Germany) mounted on an Agilent 7890A gas chromatograph system coupled to a quadrupole Agilent 5975C electron ionization mass spectrometric detector (Agilent Technologies, Palo Alto, CA, USA) equipped with a fused-silica capillary column (BP21 stationary phase; 30 m length, 0.25 mm internal diameter, and 0.25 μm film thickness) (SGE, Ringwood, VIC, Australia). The carrier gas was helium with a constant column pressure of 20.75 psi (~0.143 MPa).

The stir bars were thermally desorbed in a stream of helium carrier gas at a flow rate of 75 mL min^{-1} with the thermal desorption unit programmed from 40 to 295 °C (held 5 min) at a rate of 60 °C min^{-1} in splitless desorption mode. The analytes were focused on a programmed temperature vaporizing injector (CIS-4; Gerstel), containing a packed liner (20 mg Tenax TA), held at −10 °C with cryo cooling prior to injection. After desorption and focusing, the CIS-4 was programmed from −40 °C to 260 °C (held for 5 min) at 12 °C min^{-1} to transfer the trapped volatiles onto the analytical column. The gas chromatograph oven temperature was programmed to 40 °C (held for 2 min), raised to 80 °C (5 °C min^{-1}, held for 2 min), raised to 130 °C (10 °C min^{-1}, held for 5 min), raised to 150 °C (5 °C min^{-1}, held for 5 min), and then raised to 230 °C (10 °C min^{-1}, held for 5 min). The mass spectrometer (MS) was operated in scan acquisition (27–300 *m/z*) with an ionization energy of 70 eV. The temperature of the MS transfer line was

maintained at 230 °C. MS data acquisition was carried out in positive scan mode, although to avoid matrix interferences, the MS quantification was performed in the single ion-monitoring mode using their characteristic m/z values. Information related to analyzed compounds and m/z vales are included in greater detail in Sánchez-Gómez et al.[16] work. Compounds identification was performed using the NIST library and confirmed by comparison with the mass spectra and retention time of their pure standards (Sigma-Aldrich, Steinheim, Germany). 3-Methyl-1-pentanol was used as internal standard. Quantification was based on calibration curves of the respective standards at five different concentrations ($R^2 = 0.95$–0.97). Analyses of each replicate were performed in triplicate.

Determination of phenolic compounds by high-performance liquid chromatography with diode-array detection

The content of low molecular weight phenolic compounds of wines and SEGs extracts was determined according to the Cebrián-Tarancón et al.[10] method. For this, 20 μL of sample was injected into an Agilent 1200 (Agilent Technologies) high-performance liquid chromatography (HPLC) system equipped with a diode array detector (DAD; Agilent G1315D) coupled to an Agilent ChemStation (version B.03.01) data-processing station. Separation was performed in a reverse-phase ACE C18-PFP (4.6 mm × 150 mm, 3 μm particle size) and an ACE Excel HPLC Pre-column Filter 1PK (0.5 μm particle size) at 30 °C. The HPLC proportion of solvents used was 97.5:1.5:1 (v/v/v) water/formic acid/acetonitrile as solvent A and 78.5:1.5:20 (v/v/v) acetonitrile/formic acid/solvent A as solvent B. The elution gradient was set up for solvent B as follows: 0 min, 5%; 8.40 min, 5%; 12.50 min, 10%; 19 min, 15%; 29 min, 16%; 30 min, 16.5%; 34.80 min, 18%; 37.20 min, 32%; 42 min, 62%; 52 min, 90%; 54 min, 100%; 56 min, 100%; 60 min, 5%; 65 min, 5%. All compound detections were carried out by means of DAD by comparison with the corresponding ultraviolet–visible spectra and retention time of the compounds' pure standards (Sigma-Aldrich, Steinheim, Germany). The wavelength quantification of the compounds analyzed was outlined in greater detail in the work of Cebrián-Tarancón et al.[10] Quantification was based on the calibration curves of the respective standards at five different concentrations achieved by ultraviolet–visible signal ($R^2 = 0.92$–0.99). All analyses of each replicate were carried out in triplicate.

Statistical analysis

The descriptive analysis results regarding wine and SEGs extracts composition were examined using one-way analysis of variance at the 95% probability level, according to Tukey's test, to determine the differences between wines and SEGS extracts before and after contact. All statistical analyses were conducted using the Statgraphics Centurion statistical program (version 18.1.12; StatPoint Inc., The Plains, VA, USA).

RESULTS AND DISCUSSION

To assess the behavior of compounds in SEGs and wines, the phenolic and volatile compounds have been grouped according to their presence or not at origin in SEGs prior to contact with wine. Tables 1 and 2 show the compounds not previously found in Tempranillo and Cabernet Sauvignon SEGs and their behavior in their respective wines before and after maceration, being mostly anthocyanins. By contrast, Tables 3 and 4 show the compounds

previously found in Tempranillo and Cabernet Sauvignon SEGs and their behavior in their respective wines before and after contact, with flavanols being the most abundant and with a diversity of volatiles. To facilitate the understanding of the results, these are expressed in milligrams or micrograms in the total amount of vine shoots and volume of wine in each vinification. Furthermore, only compounds that show significant differences in wine will be discussed.

Regarding Tables 1 and 2, as expected, most of the compounds decreased their concentration in wine after SEGs–wine contact for both varieties. However, the amount retained of these compounds in wood was not proportional to the concentrations observed in wine. In the case of phenolic compounds, anthocyanins and flavonols were the families of compounds most affected by wood sorption in both varieties. Regarding flavonols, myricetin and quercetin were the compounds that showed the greatest decrease in wines, with the concentrations being 70% and 64% less respectively after contact with SEGs in the case of Tempranillo and 41% and 62% less respectively in the case of Cabernet Sauvignon wines. This reduction could be due to the different characteristics of their corresponding glycosides, which could lead to a higher affinity and retention of these compounds by the wood. In the case of anthocyanins, a different behavior was observed for each variety. In the case of Tempranillo SEGs, unlike other woods, a slight increase in the total concentration of this family of compounds was observed in wine after contact. Indeed, although most of the monomeric anthocyanins decreased their amount after contact, both malvidin-3-O-glucoside and its acetylated form showed an increase in the wine. This behavior may be due to certain anthocyanin polymeric structures present in wine that, after SEGs–wine contact, are released by the interaction with wood. Recent studies have shown that the effect of the anthocyanin/proanthocyanidin ratio is an important factor in the formation of polymeric pigments in wines.[17] Specifically, researchers observed that the production of polymeric pigments is consistently greater in the case of high anthocyanin/low proanthocyanidin concentrations, but the polymeric pigments decreased when proanthocyanidin was added to anthocyanin-containing model wines. In this line, Cebrián-Tarancón et al.,[4] reported that vine-shoots tannins are proanthocyanidins. Therefore, the SEGs addition to wines could modify the anthocyanin/proanthocyanidin ratio in wine and resulting in a reduction of these polymeric pigments. Considering that malvidin-3-glucoside is the more abundant monomeric anthocyanin, it can be thought that these polymeric structures are mainly formed by the interaction of this anthocyanin with other phenolic compounds and, therefore, that its rupture leads to an increase in the amount of malvidin-3-glucoside and its different forms. Figure 1(a) presents the HPLC-DAD chromatograms of Tempranillo wines at $\lambda = 520$ nm before and after contact with SEGs. The area comprising the hump formed by the anthocyanin polymeric pigments of wines (shown in detail) presented a further reduction of the polymeric material hump after maceration (6252.42 ± 117.52 mAU before contact and 3947.46 ± 74.63 mAU after contact), which supports the observed increase of malvidin-3-glucoside and its acetylated form in wines after Tempranillo SEGs contact.

However, Cabernet Sauvignon wines did not show a similar behavior; indeed, a decrease in all monomeric anthocyanins was observed, mainly malvidin 3-(6′-p-coumaroyl)-glucoside and petunidin-3-glucoside, with the concentrations being 36.55% and 28.81% less respectively after contact with SEGs.

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Table 1. Phenolic and volatile compounds not found in Tempranillo SEGs prior to contact with wine

| | SEGs | Wine | | | |
	After contact	Before contact	After contact	P	Δ
Phenolic compounds (mg)[a]					
Cyanidin 3-(6′-*p*-coumaroyl)-glucoside	543.08 ± 11.30	846.01 ± 29.25	819.00 ± 20.47	—	−27.40
Delphinidin 3-*O*-glucoside	998.57 ± 28.31	4 318.416 ± 2.36	3 781.78 ± 60.72	***	−536.44
Malvidin 3-*O*-glucoside	5 852.82 ± 169.75	37 380.80 ± 816.55	44 577.26 ± 63.72	***	7 196.46
Malvidin 3-(6′-acetyl)-glucoside	815.45 ± 25.09	2 962.99 ± 121.14	4 580.69 ± 1.94	***	1 617.74
Malvidin 3-(6′-cafeoil)-glucoside	816.97 ± 58.15	1 727.48 ± 65.46	1 416.14 ± 16.96	***	−312.04
Malvidin 3-(6′-*p*-coumaroyl)-glucoside	2 088.44 ± 212.03	7 218.47 ± 242.36	5 534.84 ± 9.39	***	−1 684.58
Peonidin 3-*O*-glucoside	620.48 ± 14.03	1 839.90 ± 22.25	1 712.12 ± 60.97	**	−127.38
Petunidin 3-*O*-glucoside	1 461.92 ± 37.85	7 743.87 ± 67.46	7 732.18 ± 241.63	—	−12.64
Petunidin 3-(6′-*p*-coumaroyl)-glucoside	745.85 ± 42.52	1 474.90 ± 74.71	1 282.93 ± 2.64	**	−192.10
Myricetin	297.02 ± 78.96	971.60 ± 27.43	293.21 ± 16.09	***	−678.18
Myricetin 3-*O*-galactoside	261.45 ± 10.98	218.52 ± 6.25	174.05 ± 0.55	***	−44.48
Quercetin	214.85 ± 56.88	765.57 ± 31.32	279.37 ± 14.17	***	−486.34
Quercetin 3-*O*-glucuronide + glucoside	191.15 ± 8.95	596.58 ± 3.95	414.44 ± 5.46	***	−181.84
Syringetin 3-*O*-glucoside	155.39 ± 5.17	389.27 ± 9.76	309.35 ± 0.32	***	−79.40
Coumaric acid	493.84 ± 5.09	n.d.	988.48 ± 27.96	***	988.26
Volatile compounds (mg)[a]					
Diethyl succinate	7.16 ± 0.24	49.06 ± 24.45	10.96 ± 1.31	***	−38.10
Ethyl lactate	224.25 ± 24.85	3 673.46 ± 8.59	2 906.97 ± 326.64	**	−766.47

For each compound, significant differences among wine before and after SEGs contact were examined using one-way analysis of variance according to Tukey's test (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$).
The differences Δ are calculated as 'milligrams before contact' less 'milligrams after contact'.
The mean values ($n = 6$) are shown with their standard deviation. n.d.: not detected.
[a] Phenolic and volatile compounds are expressed as milligrams of each compound in the total SEGs and volume of wine.

Table 2. Phenolic and volatile compounds not found in Cabernet Sauvignon SEGs prior to contact with wine

| | SEGs | Wine | | | |
	After contact	Before contact	After contact	P	Δ
Phenolic compounds (mg)[a]					
Delphinidin 3-*O*-glucoside	1 396.59 ± 37.43	5 017.99 ± 12.18	3 915.83 ± 4.71	***	−1 102.16
Malvidin 3-*O*-glucoside	8 172.35 ± 328.20	58 306.57 ± 300.43	46 099.58 ± 191.17	***	−12 206.99
Malvidin 3-(6′-acetyl)-glucoside	949.46 ± 41.52	3 471.75 ± 244.60	2 882.35 ± 182.68	—	−589.40
Malvidin 3-(6′-caffeoyl)-glucoside	3 612.78 ± 154.78	28 100.76 ± 162.60	22 663.47 ± 177.86	**	−5 437.29
Malvidin 3-(6′-*p*-coumaroyl)-glucoside	2 058.27 ± 157.27	6 769.27 ± 76.16	4 295.20 ± 199.52	**	−2 474.07
Peonidin 3-*O*-glucoside	1 019.25 ± 17.75	3 265.72 ± 18.19	2 581.21 ± 0.77	***	−684.50
Petunidin 3-*O*-glucoside	1 493.56 ± 41.71	6 924.49 ± 44.53	4 929.57 ± 16.49	***	−1 994.91
Petunidin 3-(6′-*p*-coumaroyl)-glucoside	787.42 ± 17.00	1 176.65 ± 72.27	1 168.17 ± 2.72	—	−8.48
Myricetin	256.33 ± 46.07	403.19 ± 18.58	236.10 ± 6.53	**	−167.09
Myricetin 3-*O*-galactoside	211.68 ± 6.93	226.62 ± 0.51	211.24 ± 0.15	***	−15.38
Syringetin 3-*O*-glucoside	219.61 ± 7.78	446.01 ± 0.11	387.44 ± 7.97	**	−58.57
Quercetin	292.55 ± 73.04	672.30 ± 41.98	254.99 ± 8.84	**	−417.32
Quercetin 3-*O*-glucuronide + glucoside	331.67 ± 19.12	1 132.95 ± 17.13	879.00 ± 5.76	**	−253.95
Coumaric acid	241.22 ± 23.66	n.d.	388.68 ± 22.68	**	388.68
Volatile compounds (mg)[a]					
Diethyl succinate	4.64 ± 0.49	10.08 ± 0.55	10.43 ± 0.83	—	0.35
Ethyl lactate	270.98 ± 12.82	7 855.93 ± 439.17	6 605.38 ± 25.78	*	−1 250.55

For each compound, significant differences among wine before and after SEGs contact were examined using one-way analysis of variance according to Tukey's test (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$).
The differences Δ are calculated as 'milligrams before contact' less 'milligrams after contact'.
The mean values ($n = 6$) are shown with their standard deviation. n.d.: not detected.
[a] Phenolic and volatile compounds are expressed as milligrams of each compound in the total SEGS and volume of wine.

**Table 3.** Phenolic and volatile compounds found in Tempranillo SEGs prior to contact with wine

	SEGs			Wine			
Compound	Before contact	After contact	P	Before contact	After contact	P	Δ
Phenolic compounds (mg)[a]							
(−)-Epicatechin	11 156.08 ± 673.24	11 832.45 ± 404.61	—	40 142.49 ± 920.28	36 326.91 ± 431.10	***	−3 815.20
Procyanidin B2	798.03 ± 27.43	643.40 ± 68.28	**	1 636.80 ± 154.81	2 353.26 ± 188.57	**	716.46
Ellagic acid	3 955.75 ± 153.09	6 633.04 ± 388.76	***	8 577.22 ± 95.28	7 508.11 ± 105.72	***	−1 079.70
trans-Resveratrol	733.30 ± 20.30	818.01 ± 218.58	—	101.02 ± 4.11	543.46 ± 49.08	***	443.38
Volatile compounds (μg)[b]							
Ethyl cinnamate	33.50 ± 2.84	86.37 ± 5.69	***	3.20 ± 0.01	20.22 ± 4.11	***	16.82
Ethyl decanoate	374.37 ± 49.75	1 310.06 ± 114.20	***	3 616.53 ± 326.40	1 866.81 ± 193.52	***	−1 748.68
Ethyl hexanoate	84.53 ± 6.39	58.74 ± 10.16	***	2 614.83 ± 178.35	5 547.49 ± 1678.73	**	2 932.96
Ethyl octanoate	5 167.36 ± 782.97	22 333.13 ± 2181.04	***	994 763.66 ± 73 424.39	1 722 644.35 ± 517 080.74	*	727 881.36
2-Phenylethyl acetate	45.10 ± 2.81	364.73 ± 43.75	***	820.01 ± 40.08	924.06 ± 68.43	*	103.76
Benzyl alcohol	3 708.31 ± 624.17	9 517.76 ± 2342.87	***	10 732.70 ± 372.15	13 373.10 ± 302.32	***	2 640.56
1-Hexanol	2 172.03 ± 52.89	3 477.64 ± 224.98	***	62 007.57 ± 2821.06	112 330.74 ± 9613.09	***	50 322.40
Nonanol	267.35 ± 15.70	575.04 ± 109.31	***	273.27 ± 42.99	268.15 ± 0.33	—	−6.06
Phenylethyl alcohol	6 469.88 ± 438.66	324 115.36 ± 63 654.81	***	290 660.99 ± 11 659.36	324 283.17 ± 19 795.79	*	33 621.34
Decanoic acid	7 363.37 ± 721.23	118 595.99 ± 51 988.79	***	18 488.37 ± 7.39	5 576.44 ± 1129.68	***	−12 911.52
Hexanoic acid	63 569.42 ± 3263.58	58 607.07 ± 6603.56	—	109 688.88 ± 8525.69	248 413.62 ± 27 191.11	***	138 725.62
Octanoic acid	9 434.00 ± 288.67	123 774.19 ± 14 993.44	***	97 579.43 ± 5282.72	149 079.73 ± 17 934.05	***	51 500.64
Benzaldehyde	1 323.19 ± 62.15	1 337.53 ± 248.54	—	280.40 ± 35.52	603.56 ± 198.14	**	323.16
Nonanal	1 017.76 ± 68.87	2 280.66 ± 250.38	***	166.40 ± 26.22	250.30 ± 86.95	—	83.90
Guaiacol	522.38 ± 19.90	473.86 ± 204.74	—	2 431.84 ± 79.72	2 997.67 ± 352.73	**	565.83
Vanillin	13 254.30 ± 1714.50	17 468.08 ± 3305.44	*	n.d.	61.35 ± 4.20	***	61.35
Geraniol	n.d.	n.d.	—	914.76 ± 12.38	1 115.40 ± 29.27	***	200.64
Geranyl acetone	78.40 ± 8.66	55.79 ± 11.11	**	n.d.	n.d.	—	n.d.
β-Ionone	81.42 ± 3.50	84.23 ± 7.20	—	40.00 ± 0.02	54.60 ± 1.82	***	16.42

For each compound, significant differences among wine before and after SEGs contact were examined using one-way analysis of variance according to Tukey's test (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$).
The differences Δ are calculated as 'milligrams before contact' less 'milligrams after contact'.
The mean values ($n = 6$) are shown with their standard deviation. n.d.: not detected.
[a] Phenolic compounds are expressed as milligrams of each compound in the total SEGs and volume of wine.
[b] Volatile compounds are expressed as micrograms of each compound in the total SEGs and volume of wine.

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Table 4. Phenolic and volatile compounds found in Cabernet Sauvignon SEGs prior to contact with wine

Compound	SEGs			Wine			
	Before contact	After contact	P	Before contact	After contact	P	Δ
Phenolic compounds (mg)[a]							
(−)-Epicatechin	15 360.19 ± 512.91	22 318.27 ± 766.54	***	70 327.49 ± 142.29	89 108.75 ± 855.92	**	18 781.27
Procyanidin B2	804.02 ± 251.63	651.68 ± 69.26	—	1 825.18 ± 84.35	3 546.11 ± 785.58	—	1 720.93
Ellagic acid	4 712.28 ± 19.80	7 695.09 ± 366.55	***	9 140.38 ± 123.58	8 806.30 ± 19.03	*	−334.08
Piceatannol	322.01 ± 29.79	435.99 ± 102.32	*	n.d.	n.d.	—	n.d.
trans-Resveratrol	735.22 ± 147.90	1 285.87 ± 307.49	*	153.31 ± 5.98	528.34 ± 78.97	*	375.03
Volatile compounds (μg)[b]							
Ethyl cinnamate	48.57 ± 8.05	205.93 ± 27.71	***	16.69 ± 0.65	16.42 ± 1.38	—	−0.27
Ethyl decanoate	348.16 ± 21.32	1 500.59 ± 79.10	***	3 879.79 ± 144.70	3 388.44 ± 271.44	—	−491.35
Ethyl hexanoate	171.01 ± 11.11	96.44 ± 6.38	***	4 672.83 ± 32.32	6 344.75 ± 492.17	**	1 671.92
Ethyl octanoate	8 390.06 ± 782.28	25 184.85 ± 1115.70	***	3 780 456.72 ± 123 152.60	4 399 400.06 ± 416 485.08	—	618 943.34
2-Phenylethyl acetate	52.38 ± 14.95	948.15 ± 87.32	***	4 314.56 ± 126.75	3 586.74 ± 334.52	*	−727.83
1- Hexanol	3 814.67 ± 331.52	3 596.37 ± 389.79	—	94 079.34 ± 1813.72	148 311.64 ± 2451.39	***	54 232.30
Nonanol	435.92 ± 35.98	904.24 ± 72.55	***	161.01 ± 14.43	362.86 ± 23.03	***	201.85
Benzyl alcohol	3 086.28 ± 271.01	7 253.12 ± 507.68	***	23 891.81 ± 400.71	28 970.97 ± 1299.13	**	5 079.16
Phenylethyl alcohol	6 929.29 ± 977.73	583 361.15 ± 40 697.11	***	1 094 861.27 ± 26 854.56	1 110 151.89 ± 79 560.92	—	15 290.63
Octanoic acid	13 795.76 ± 1643.83	95 289.81 ± 14 545.64	***	142 354.59 ± 11 448.11	149 919.00 ± 17 699.14	—	7 564.41
Decanoic acid	13 973.88 ± 355.68	59 701.25 ± 10 306.29	***	18 790.66 ± 1833.50	13 876.57 ± 1059.76	*	−4 914.09
Hexanoic acid	50 347.07 ± 2618.94	29 634.52 ± 2591.67	***	161 587.80 ± 23 425.04	204 762.34 ± 87 119.10	—	43 174.54
Nonanal	2 014.29 ± 285.60	2 033.10 ± 671.39	—	258.47 ± 3.41	293.19 ± 12.09	**	34.72
Benzaldehyde	2 498.94 ± 561.49	1 648.99 ± 304.24	*	432.62 ± 40.46	608.14 ± 52.16	*	175.52
Guaiacol	253.26 ± 164.37	705.75 ± 83.93	**	2 241.47 ± 362.57	2 615.84 ± 118.52	—	374.37
Vanillin	n.d.	n.d.	—	n.d.	n.d.	—	0
Geraniol	n.d.	n.d.	—	1 045.28 ± 39.22	1 180.51 ± 31.31	**	135.23
Geranyl acetone	122.29 ± 5.94	60.37 ± 0.91	***	n.d.	n.d.	—	n.d.
β-Ionone	114.21 ± 16.72	95.84 ± 12.72	*	56.46 ± 0.16	67.56 ± 0.94	***	11.11

For each compound, significant differences among wine before and after SEGs contact were examined using one-way analysis of variance according to Tukey's test (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$).
The differences Δ are calculated as 'milligrams before contact' less 'milligrams after contact'.
The mean values ($n = 6$) are shown with their standard deviation. n.d.: not detected.
[a] Phenolic compounds are expressed as milligrams of each compound in the total SEGs and volume of wine.
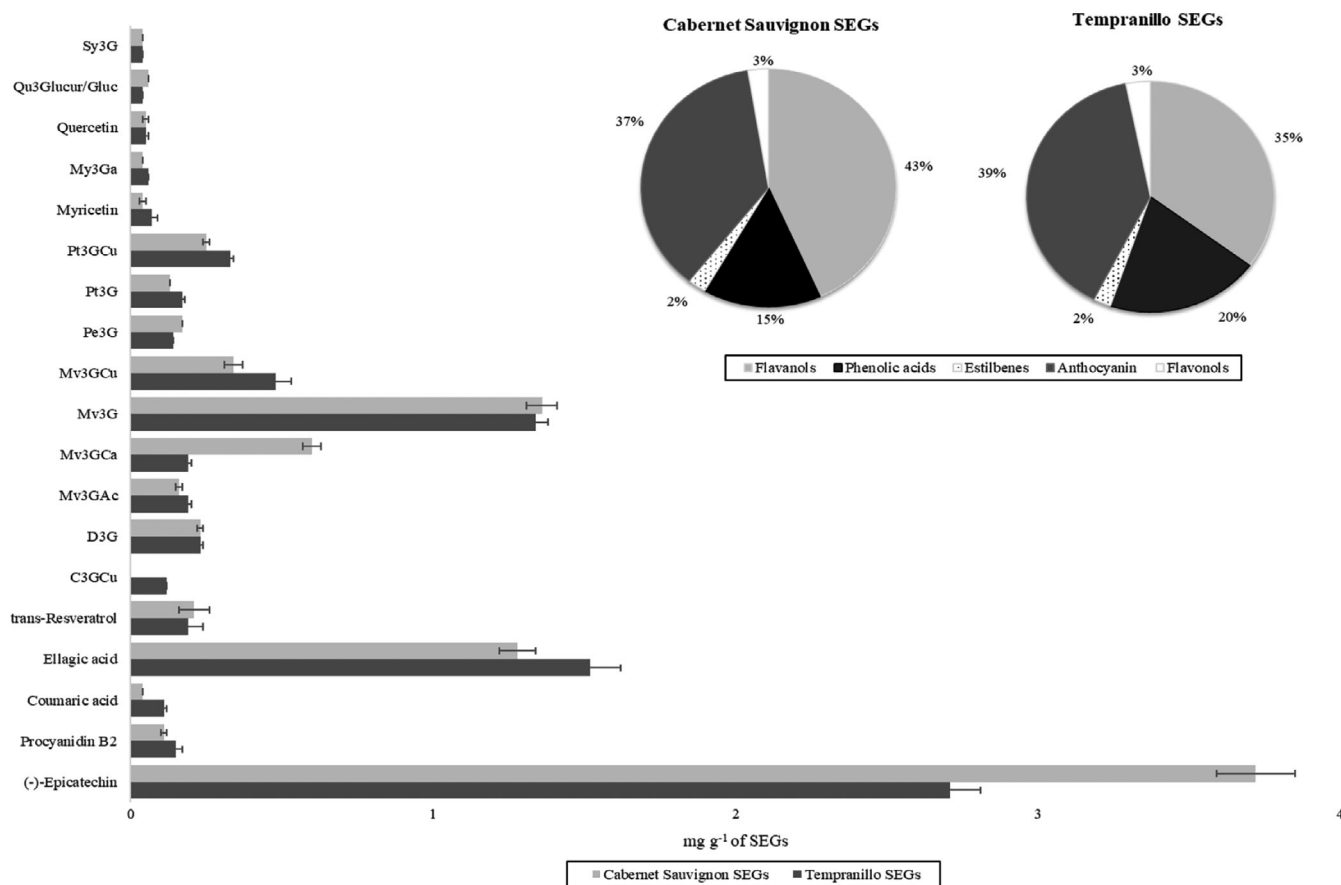[b] Volatile compounds are expressed as micrograms of each compound in the total SEGs and volume of wine.

(a)

(b)
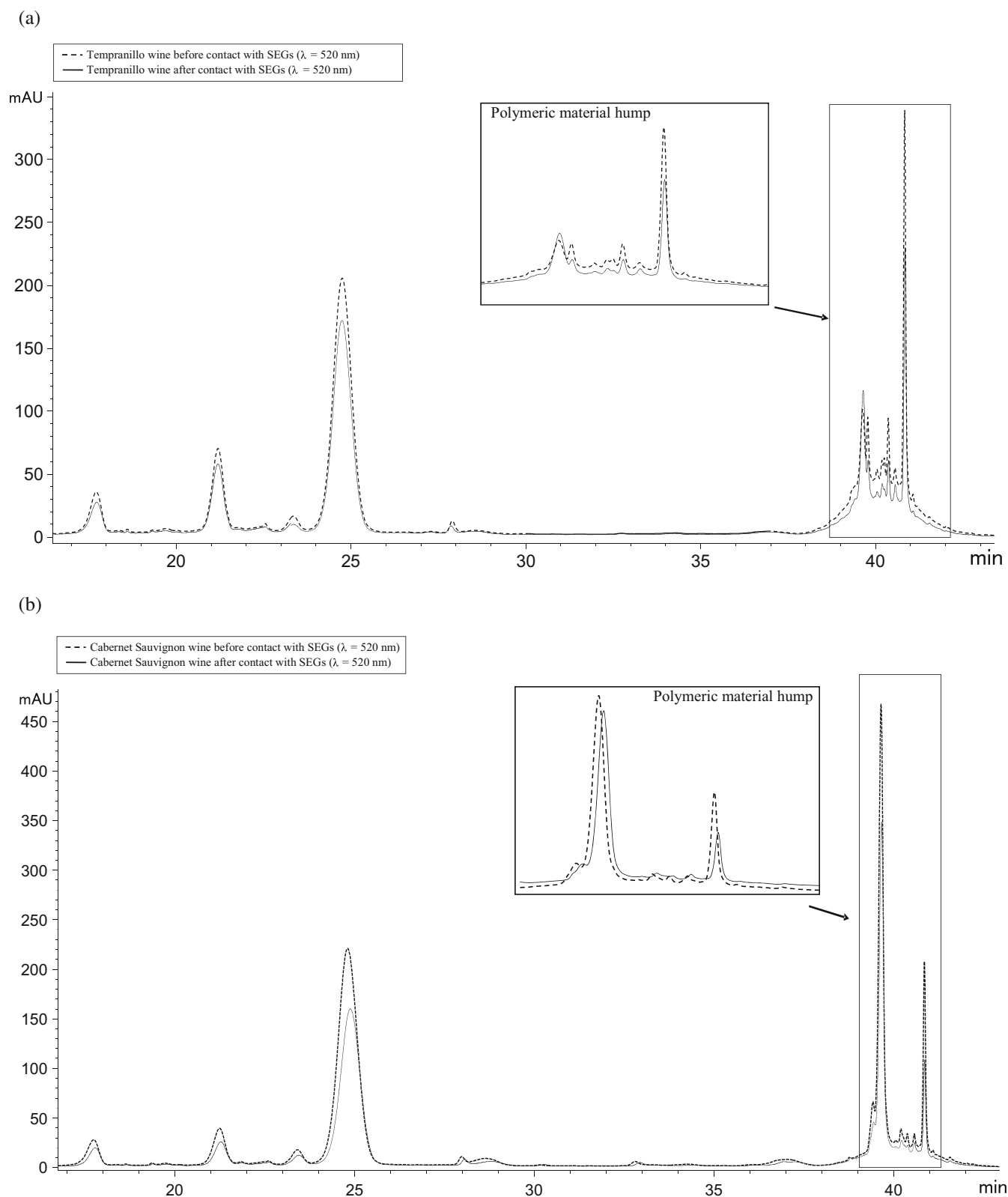
Figure 1. Chromatograms with anthocyanin peaks and polymeric material hump detail for wines before and after contact with SEGs: (a) Tempranillo; (b) Cabernet Sauvignon.

This agrees with its HPLC-DAD chromatogram (Fig. 1(b)), where the reduction in the polymeric material hump was lower: 2713.09 ± 55.36 mAU before contact and 2356.12 ± 117.88 mAU after contact.

Some studies carried out with other woods (oak, acacia, and cherry) pointed out that each type of wood has a particular extraction kinetics.[18] In this case, although both are vine-shoot woods, two different varieties have been considered, and

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Figure 2. Phenolic composition of SEGs (mg g^{-1}) after contact with wine. (a) Percentage of each phenolic family in relation to the total phenolic compounds content. (b) Individual phenolic compounds. Sy3G: syringetin 3-*O*-glucoside; Qu3Glucur/Glu: quercetin 3-*O*-glucuronide + quercetin 3-*O*-glucoside; My3Ga: myricetin 3-*O*-galactoside; Pt3GCu: petunidin 3-(6′-*p*-coumaroyl)-glucoside; Pt3G: petunidin 3-*O*-glucoside; Pe3G: peonidin 3-*O*-glucoside; Mv3GCu: malvidin 3-(6′-*p*-coumaroyl)-glucoside; Mv3G: malvidin 3-*O*-glucoside; Mv3GCa: malvidin 3-(6′-*t*-caffeoyl)-glucoside; Mv3GAc: malvidin 3-*O*-(6′-acetyl)-glucoside; D3G: delphinidin 3-*O*-glucoside; C3GCu: cyanidin 3-(6′-*p*-coumaroyl)-glucoside.

therefore their chemical composition[9,19] and physical properties could be sufficiently different to justify the variability of total and individual extractable phenolic compounds between the two SEGs varieties.

Coumaric acid was the only phenolic acid detected in SEGs after contact, with amounts of 241.22 mg in Cabernet Sauvignon and 493.84 mg in Tempranillo. This compound was not detected in wines before maceration, showing a similar behavior in both varieties. The increase of this compound in wine can be explained by a release, favored by maceration with wine (ethanolic solution), from the units of coumaroyl monolignols of vine-shoots lignin.[5,20]

Focusing on volatile compounds not found in SEGs before contact (Tables 1 and 2), only diethyl succinate and ethyl lactate were detected in both cases. These compounds are not usually found in vine-shoot wood, and their presence is clearly a consequence of wood extraction from wine composition during the contact, which has also been reported in other studies.[14] It should be noted that, although the amount of ethyl lactate was higher in Cabernet Sauvignon than in Tempranillo, the amount absorbed by SEGs was very similar for both varieties, suggesting a possible absorption saturation point of this compound for vine shoots. This agrees with Coelho *et al.*,[14] who suggested that the maximum sorption capacity depends mainly on the type of wood and not on the size of the chips.

On the other hand, Tables 3 and 4 show the phenolic and volatile compounds determined in SEGs before contact, as well as

their behavior in SEGs and wine after that. The phenolic compounds found above the quantification limit were (−)-epicatechin, procyanidin B2, ellagic acid, and *trans*-resveratrol. Ellagic acid was the most retained compound and showed a similar trend in both varieties, increasing its content in SEGs and decreasing in wines. However, although the behavior in wood was very similar in both varieties, the decrease in wine was greater in Tempranillo than in Cabernet Sauvignon (12.59% and 3.65% respectively), which could be due to some possible precipitation of this compound, as previously observed by others,[21] or due to their involvement in the anthocyanins transformation.[22]

(−)-Epicatechin showed a different trend between varieties. In the case of Tempranillo, this compound decreased by 9.50% in wine, whereas the differences observed in SEGs wood before and after contact were not significant. However, in Cabernet Sauvignon, the amount of (−)-epicatechin increased significantly in SEGs and wine with the maceration (45.30% and 26.71% respectively). This behavior confirms the varietal character that this compound shows in vine-shoots, as Cebrián-Tarancón *et al.*[19] suggested when a classification of the vine shoots according to their enological aptitude was carried out. In addition, Cabernet Sauvignon wood has a higher concentration of (−)-epicatechin than Tempranillo wood does according to Cebrián-Tarancón *et al.*[9] Hence, during Cabernet Sauvignon SEGs maceration, a greater release of (−)-epicatechin from the wood into the wine
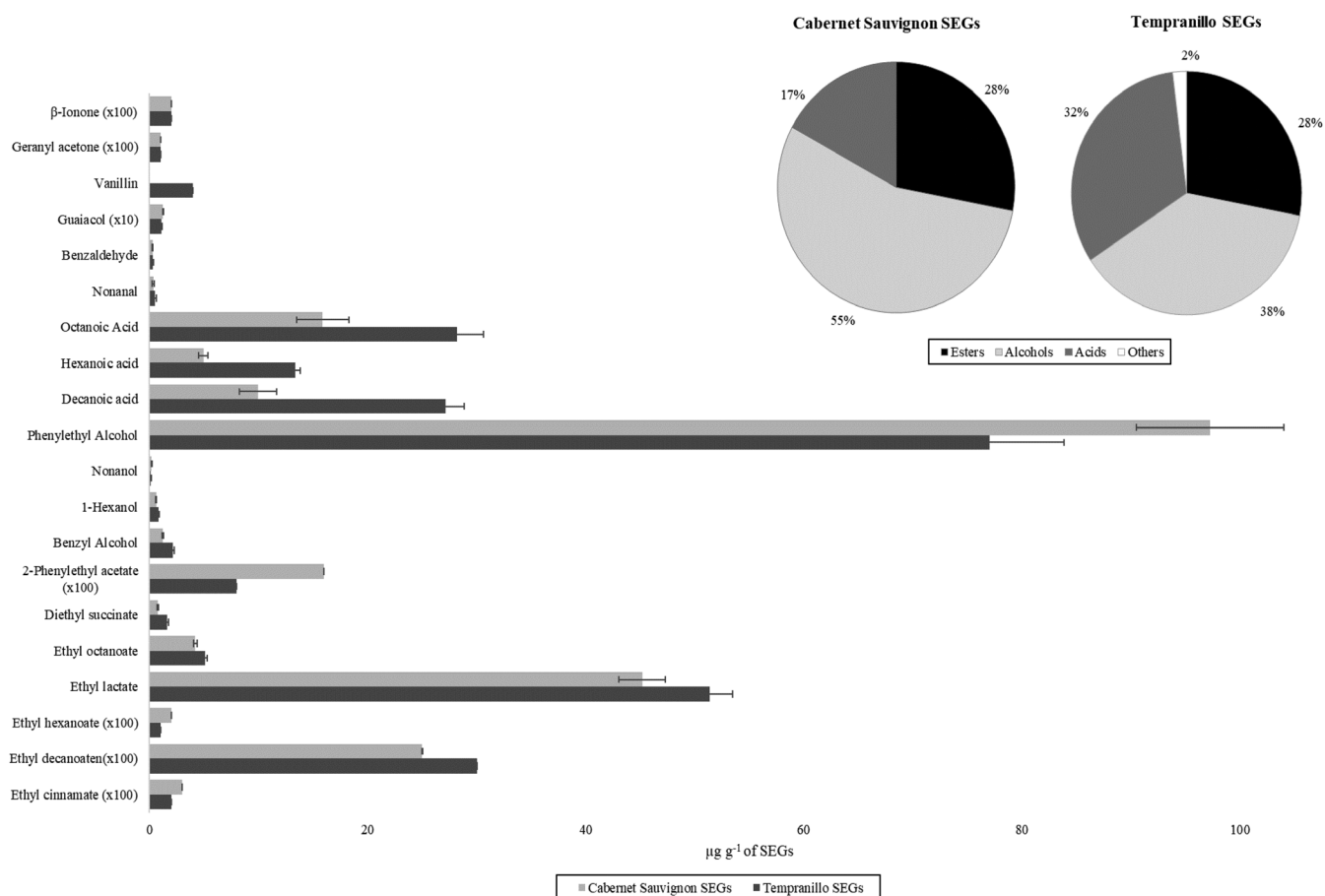
Figure 2 (Continued)

takes place. Procyanidin B2 had a similar trend in both varieties, showing a decrease in SEGs after contact; this was reflected in an increase in the wines, this being more significant in Cabernet Sauvignon.

It is well known that vine shoots are an important source of stilbenes, mainly *trans*-resveratrol.[3,23] Therefore, the increase of this compound in the wines after maceration with SEGs was expected. However, the increase in *trans*-resveratrol after contact with wine was not associated with a proportional decrease in SEGs, but with an increase in both varieties. This could be justified by the fact that the 30 days of SEGs contact with the wine produces a degradation of the lignocellulosic framework that favors the accessibility of this compound, facilitating its subsequent extraction. Therefore, the SEGs after their use in wine as enological additives could continue to be an important source of *trans*-resveratrol and be reused for different purposes.

Regarding volatile compounds, phenylethyl alcohol was the compound that increased more in SEGs after contact, but this increase was not very significant in the case of Tempranillo wines and showed no difference in Cabernet Sauvignon wines. Despite this compound also being previously detected in vine shoots by other authors,[3] its high quantity in SEGs after contact is a clear consequence of wood sorption during the contact period and can be associated with the high polarity of this compound. This fact was also reported by other researchers with oak wood.[14,24,25] This behavior is consistent with the results recently obtained by Cebrián-Tarancón *et al*.[12] where an increase of phenylethyl

alcohol was observed in wine when SEGs were added after alcoholic fermentation, but it remained constant in wines when SEGs were added after malolactic fermentation and the time of contact was higher. Benzyl alcohol also increased significatively in SEGs and wines of both varieties after contact, showing a similar trend. Considering the rest of the alcohols, it was not possible to define a standard behavior for 1-hexanol and nonanol since the trend was different depending on the variety. In the case of Tempranillo, the amount of both compounds increased in SEGs, but in wine only 1-hexanol showed differences, with an increase of 81.16%. On the other hand, in Cabernet Sauvignon, an increase of both compounds was found in wines but only nonanol showed differences in SEGs, increasing its amount to 107.43%.

Regarding esters, phenylethyl acetate and ethyl decanoate were significantly retained by SEGs in both varieties. However, in wines, the content of the former remained similar before and after maceration and the latter decreased, which suggests adsorption by the wood.

A correlation was observed between the chain length of the lipophilic portion and sorption, contrary to what was observed by Ramirez *et al*.[24] for oak wood. Whereas ethyl hexanoate and hexanoic acid decreased their sorption on SEGs after contact, the sorption of ethyl octanoate and ethyl decanoate and their respective acids was markedly increased. The behavior exhibited by these compounds could be associated with the different size and morphological characteristics of the SEGs fibers,[26,27] which

could result in their different properties and, therefore, in a higher coupling or affinity for such compounds with a larger molecule size.

In terms of volatile phenols, only guaiacol and vanillin were detected. The former was found in both varieties, although it showed a different behavior between them. In the case of Tempranillo SEGs, it remained constant, but in wines it increased after contact, which is clearly due to a release of part of the wood from the vine shoots. However, in Cabernet Sauvignon, the opposite behavior was observed, since the content of guaiacol increased in SEGs after contact and remained constant in wines. In a similar way to other compounds, this behavior suggest a varietal character of this compound in vine shoots. Cabernet Sauvignon SEGs are richer in guaiacol than Tempranillo SEGs are (Tables 3 and 4). However, guaiacol is easily accessible in the latter, resulting in a better release of guaiacol from vine shoots to wine. Vanillin was only detected in Tempranillo, although it was only found in wine after its contact with SEGs, which agrees with the results recently obtained by Cebrián-Tarancón *et al.*[12] The presence of vanillin in Tempranillo toasted vine shoots has been previously described in other studies and is associated with the degradation of the lignin structure during the toasting process,[3,5] so the release of vanillin from vine shoots to the wines was expected. The slight increase ($P < 0.05$) of this compound in SEGs after contact can be associated, as described earlier herein for *trans*-resveratrol, to an increase in the accessibility of this within the vine-shoots' fibers after contact with wine.

Geraniol and geranyl acetone showed similar behavior in both varieties. The former was only detected in wine and increased after contact, whereas the latter was only detected in SEGs and decreased after contact. The transformation of geraniol to geranyl acetone takes place through an esterification reaction.[28]

Possible applications of SEGs after wine contact

Once SEGs have been in contact with the wine, their chemical composition is modified due to the sorption or release of the compounds described earlier herein, which could increase their interest for possible new uses. Figure 2 shows the phenolic and volatile compositions of Tempranillo and Cabernet Sauvignon SEGs after contact with wine either by chemical families or individually, in order to highlight those compounds with the greatest presence.

In Tempranillo and Cabernet Sauvignon SEGs, the phenolic composition (Fig. 2) accounts for 97.44% and 97.99% respectively of the total phenolic and volatile compounds determined, where the main families were flavanols, anthocyanins, and phenolic acids. Flavanols were the first group of compounds in Cabernet Sauvignon SEGs, but were second in the case of Tempranillo SEGs. However, the main flavanol in both varieties was (−)-epicatechin, with levels of 2.71 mg g^{-1} (95% of the total flavanols) and 3.72 mg g^{-1} (97% of the total flavanols) for Tempranillo and Cabernet Sauvignon respectively, whose antioxidant effect are well known.[29] Moreover, phenolic acids comprised 20% in Tempranillo SEGs and 15% in Cabernet Sauvignon SEGs, with ellagic acid being the most important, with levels of 1.52 mg g^{-1} (93% of the total phenolic acids) and 1.28 mg g^{-1} (97% of the total phenolic acids) for Tempranillo and Cabernet Sauvignon respectively. This compound has influence not only on the color, astringency, and bitterness of wines, but also on the antioxidant capacity.[30] Hence, the vine shoots, after being used as additives, have a high amount of such phenolic compounds, which suggests their use as an antioxidant product.

The retention of anthocyanins by SEGs was also very significant, accounting for 37% and 39% in Tempranillo SEGs and Cabernet Sauvignon SEGs respectively of the total phenolic compounds. The most abundant monomeric anthocyanin was malvidin-3-*O*-glucoside, with a concentration close to 1.40 mg g^{-1} in both varieties (42% of the total anthocyanins), followed by malvidin 3-acetyl-glucoside and malvidin 3-caffeoyl-glucoside (Supporting Information Table S3). The fact that vine shoots retain a high quantity of anthocyanins during their contact with wine makes them a potential source of these compounds, which are highly valued as food colorants or as nutraceutical ingredients, as they provide numerous beneficial health effects.[31]

The volatile composition of SEGs was significantly modified after contact with wine. The most abundant family of compounds were alcohols, which accounted for 38% and 55% of the total volatile compounds in Tempranillo SEGs and Cabernet Sauvignon SEGs respectively. The most abundant compound was phenylethyl alcohol, whose concentration was 77.03 mg g^{-1} (96% of the total alcohols) and 97.23 mg g^{-1} (98% of the total alcohols) for Tempranillo and Cabernet Sauvignon respectively. Esters had a similar amount in both varieties, 28% of the total volatile compounds, with ethyl lactate being the main one with concentrations of 51.34 mg g^{-1} and 45.16 mg g^{-1} for Tempranillo and Cabernet Sauvignon varieties respectively, reaching 24% and 25% of the total volatile compounds respectively. Compared with the phenolic composition, the volatile composition of the SEGs does not exceed 4%, and therefore the recovery of such compounds does not seem to be of interest. However, it is known that pruned vine shoots have been used to obtain lactic acid[32] from hemicellulose. Since our results have shown that the use of SEGs in wines significantly increases the ethyl lactate content, the SEGs after being used could still be a source of lactic acid but also ethyl lactate, which, by a simple hydrolytic process, would produce lactic acid.

CONCLUSIONS

Pruned vine shoots used as enological additives (SEGs) transfer some compounds to the wine that give it distinctive characteristics appreciated by tasters. However, they also retain wine compounds (especially anthocyanins from red wine) without compromising wine quality. Sorption differences have been observed depending on the variety of SEGs used. Specifically, some malvidin-derived anthocyanins retained by SEGs were increased in Tempranillo wines but not in Cabernet Sauvignon wines. Varietal behavior was also observed in flavanols, which are the most abundant compounds in all SEGs before contact with wine, increasing (−)-epicatechin in both Cabernet Sauvignon SEGs and wine and decreasing in Tempranillo wine. It should be noted that *trans*-resveratrol, present in SEGs prior to use, was transferred to wines, but was also increased in SEGs regardless of variety. Therefore, the high content of phenolic compounds in SEGs after their use as enological additives in red wines suggests that they can be reused as raw materials to obtain anthocyanins and important antioxidants, especially *trans*-resveratrol.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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