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Impact of French Oak Chip Maturation on the Volatile Composition and Sensory Profile of Agiorgitiko Wine

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Abstract: The traditional practice of aging wines in oak barrels has long been associated with the evolution of wine aromas. However, due to rising costs, alternative approaches like aging with oak chips have gained popularity. The aging time, addition dose, and type of toasting of the oak chips are critical parameters affecting the quality of the wine's aroma. In this study, we focus on wines from Agiorgitiko variety and explore the impact of oak chip maturation on both volatile composition and sensory profile. By analyzing volatile compounds of wine aroma using GC-MS/MS and conducting descriptive sensory analysis, we investigate the effects of three different oak chip toasting levels, three dosages, and three aging periods. Our findings reveal that almost all wines aged with oak chips exhibit higher ester concentrations compared to the control. Notably, heavily toasted oak chips contribute to the sensory attribute of smoky aroma, while medium oak chips are associated with the sensory attribute of barrel aroma. This study provides valuable data for winemakers to determine the most suitable application for their product.

Keywords: Agiorgitiko; Nemea; oak chips; sensory; GC-MS/MS

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1. Introduction

The aging process significantly influences the evolution and quality of wines, with the most common method of aging involving the use of oak barrels, which profoundly affects the wine's composition. This has been explored in numerous studies, such as the one by Garde-Cerdán et al., which investigated the impact of wine composition, aging duration, geographic origin, and oak barrel type on the accumulation of oak compounds in red wines aged in oak barrels [1]. Another study compared new barrels with once-used barrels, focusing on the extraction and evolution of volatile oak wood compounds [2], while Perez-Prieto et al. examined how the oak origin, barrel volume, and age affect the volatile composition of red wine after six months of maturation [3]. The origin of the oak wood from which the barrel was created [4] and the volume of the barrel [5] have also been explored, with the abundance of studies on barrel aging evident from a related review [6].

In addition to these studies, researchers have examined the extraction kinetics of volatile compounds associated with barrel oak. P.J. Spillman et al., for instance, explored the extraction of oak volatiles using model wine in new oak barrels over a two-year period [7]. Other investigations have focused on the extraction kinetics of volatile oak compounds and esters [8], or vanillin specifically [9]. Recognizing the need to utilize data on the kinetic extraction of volatile compounds of wine aroma and predict changes in the composition of wine aging in barrels, studies have aimed to determine the existence of patterns [10] or to develop a model to apply it to the study of the extraction of aroma compounds [11].

In response to the high cost of traditional oak barrel aging, new techniques have emerged, one of which involves aging wine using oak wood pieces. This method, approved by the International Organization of Vine and Wine (OIV), empowers winemakers to use appropriately sized wood pieces from *Quercus* species, which can be grilled or burnt but not charred, with the quantity of oak wood pieces left to the winemaker's discretion [12]. In Europe, this practice was first recognized as an approved oenological practice in 2006, according to Council Regulation (EC) No 2165/2005 [13], and is currently governed by Commission Delegated Regulation (EU) 2019/934 [14].

The emergence of new aging technology using wood chips has prompted extensive research on wines aged in barrels compared to those with added oak chips [15–18]. These comparative studies extend beyond barrel aging and encompass other techniques, including oak staves [19], vine-shoots [20,21], and micro-oxygenation [22]. A brief mention of micro-oxygenation is warranted, as aging in barrels facilitates the gradual incorporation of oxygen, significantly influencing the development of phenolic compounds and color stabilization [22]. In contrast, this effect is not observed to the same extent with the use of oak wood chips, primarily due to the shorter application period. In a comprehensive review, Yang Tao et al. summarized the multitude of comparative studies related to wine aging technologies [23].

In addition to comparative tests between aging technologies, there are also numerous studies focusing exclusively on oak chips. A.M. Martínez-Gil et al. conducted comparative tests on oak chips from different geographical origins [24], while M. Tavares et al. compared the impact of cherry, acacia, and oak chips [25].

Most studies on wine aging with oak chips, a critical factor for the resulting wine quality, focus on the contact time of the wood with the wine [26–28]. The dosage of oak chips, which significantly influences the wine's character, has also been a particular point of interest for researchers, as evidenced by B. Gordillo et al.'s investigation into the impact of applying two proportions of oak chips [28]. Another essential parameter is the toasting level. B. Fernandez de Simon found that the toasting intensity affects the volatile composition more than the choice of seasoning method, whether traditional or unconventional [19], a finding further supported by D.I. Stegarus et al., who emphasized that the volatile compounds extracted from the wood depend on both the degree of toasting and the duration of contact with the wine [16]. In an effort to optimize chip dosage and maceration time, B. Gordillo et al. conducted research using two different doses of oak chips at two maceration times [28]. Of particular interest is the comparison of experimental wines with commercial wines regarding their volatile and sensory profiles [29].

The grape variety from which the wine originates is another crucial parameter, a fact underscored by A. Baiano et al.'s study on the effect of treating Aglianico and Montepulciano, two different wine varieties, with oak chips. Their conclusion that the impact of oak chips depends on the grape variety [30] aligns with the findings of J. Laqui-Estaña et al., who investigated the impact of variety on phenolic composition during aging and observed significant differences between different cultivars under the same aging conditions [15]. These conclusions collectively underscore the necessity of studying each variety of interest separately, given the uncertainty of transferring research results to other varieties.

The Agiorgitiko variety, one of the most widespread wine grape varieties in Greece and renowned for its versatility, is particularly associated with Protected Designation of Origin (PDO) Nemea wines. Despite this, there are few studies on its aging process using wood chips. One such study was conducted by Koussissi et al., where they focused on red wine made from the Agiorgitiko grape. The wine in their study was aged in stainless steel tanks, either without wood or with oak chips at three different toasting levels, and was then compared with wine aged in an American oak barrel. The entire aging process lasted 32 days [31].

In relation to the Agiorgitiko variety, another study conducted by Maria Kyraleou et al. investigated the differentiation of wines treated with wood chips [32]. They used five different types of wood chips: American, French, Slavonian, Acacia, and a mixture of American and French oak. These wood chips were applied for a period of three months, with all types of wood chips being of one toasting type, and the addition was in a single dose. The study concluded that the effect of wood chips on the produced wine depends not only on the type of wood but also on the contact time with the wine.

The present study aims to expand upon the existing studies on Agiorgitiko, focusing on its aging with the addition of wood chips. For this purpose, international literature and parameters studied in wines from other grape varieties were taken into account. The study employed oak wood, the most widespread type of wood chips in the industry, and considered variables such as the type of toasting, the dosage, and the contact time. Three types of toasting were used (low, medium, and heavy), along with three doses of oak chips, over a period of three months. The kinetics of volatile compounds of wine aroma were studied monthly, and analyses were performed to determine volatile compounds of wine aroma using gas chromatography/mass spectrometry/mass spectrometry (GC-MS/MS) [33]. Sensory analysis was conducted according to methods developed by the authors [34].

The aim of this study is not only to explore the aging of the Agiorgitiko variety and compare the results with studies on wines from other grape varieties, but also to compare the experimental wines with commercial wines PDO Nemea, which have been analyzed in previous studies by the authors [33,34]. Ultimately, the study aims to provide practical information to oenologists and winemakers for optimal utilization based on their product goals.

2. Materials and Methods

2.1. Wines

For the realization of the present study, red wine from the Agiorgitiko variety (*Vitis vinifera* L.) was utilized. The wine was produced in a winery during the 2017 harvest, following a red vinification protocol. Malolactic fermentation occurred after the completion of alcoholic fermentation.

2.2. Oak Chips

The wood chips used were commercial products sourced from French oak (*Quercus robur*), provided by the company Oak Add-Ins Nadalié (Ludon-Médoc, France). Specifically, for this study, three types of French oak chips were employed, each with a different level of toasting: low toasted (LT), medium toasted (MT), and heavy toasted (HT). These chips were irregular fragments, approximately 1 cm × 1.5 cm × 0.2 cm in size. The weight for each level of toasting was calculated using the following methodology. The weight of 100 pieces was measured in triplicate. The weights recorded were as follows: low toasted, 120 ± 19 g/100 pieces; medium toasted, 54 ± 7 g/100 pieces; and high toasted, 33 ± 6 g/100 pieces.

2.3. Samples' Creation—Experimental Design

The original wine was divided into batches of approximately 20 L. Two batches remained without the addition of oak chips and served as the control group. The remaining batches were supplemented as follows: (b) Low Toasted: 1 g/L ("LT_1g"), (c) Low Toasted: 2 g/L ("LT_2g"), (d) Low Toasted: 4 g/L ("LT_4g"), (e) Medium Toasted: 1 g/L ("MT_1g"), (f) Medium Toasted: 2 g/L ("MT_2g"), (g) Medium Toasted: 4 g/L ("MT_4g"), (h) Heavy Toasted: 1 g/L ("HT_1g"), (i) Heavy Toasted: 2 g/L ("HT_2g"), (k) Heavy Toasted: 4 g/L ("HT_4g"). The addition of oak chips was performed in duplicate, and the samples were enclosed in a bag-in-box. Notably, different containers were used for the three contact periods.

2.4. Chemicals and Reagents

For the analysis of aromatic volatile compounds via gas chromatography/mass spectrometry/mass spectrometry (GC-MS/MS), high-purity reference standards were sourced from various commercial suppliers. The compounds utilized included 2-methoxy-4-methylphenol, 2-methoxy-4-vinylphenol, 2-phenylethanol, 3-(methylthio)propionaldehyde, 4-ethylguaiacol, 4-vinylphenol, acetovanilone, benzyl acetate, citral, damascenone, ethyl 3-hydroxybutyrate, ethylcapoxybutyrate, hexanal, isoeugenol, thymol, and whiskey lactone from Sigma Aldrich (St. Louis, MO, USA). Additionally, 2-phenethyl acetate, ethyl butyrate, ethyl isobutyrate, ethyl isovalerate, hexyl acetate, isobutyl acetate, rose oxide, and β-ionone were obtained from Honeywell Fluka (Charlotte, NC, USA). Furthermore, compounds such as 4-ethylphenol, citronellol, decylaldehyde, ethyl 2-methylbutyrate, ethyl caproate, ethyl trans-cinnamate, ethylvanillin, eugenol, geraniol, isoamyl acetate, linalool, and vanillin were acquired from Acros Organics (Geel, Belgium). A comprehensive list of these standards, including their Chemical Abstracts Service (CAS) Numbers, molecular formulas, purities, and suppliers, is detailed in Table A1.

For the extraction of volatiles, the following reagents were employed: water (for UHPLC, supergradient) and dichloromethane (99.8%, for pesticide analysis) sourced from PanReac AppliChem ITW Reagents (Barcelona, Spain). Ammonium sulfate ($(NH_4)_2SO_4$), sodium sulfate (Na_2SO_4), and phenol (≥99.5%) were procured from Penta Chemicals Unlimited (Prague, Czech Republic). Absolute ethanol (≥99.8%) was obtained from Honeywell (Charlotte, NC, USA), while tartaric acid (99.5%) was sourced from Sigma Aldrich (St. Louis, MO, USA).

2.5. Physicochemical Analyses

The initial wine, used both as a control and as a matrix for the addition of oak chips, underwent physicochemical analyses following methods established by the International Organization of Vine and Wine (OIV) [35] and validated, accredited procedures [36]. Specifically, the following analyses were conducted in duplicate. Alcoholic Strength, using a hydrostatic balance Gibertini (Milan, Italy) according to OIV-MA-AS312-01A. Density at 20 °C, using a hydrostatic balance according to method OIV-MA-AS2-01A. pH using pH-meter according to method OIV-MA-AS313-15, total acidity according to the titrimetric method OIV-MA-AS313-01, volatile acidity using electronic titrator "Quick" Gibertini (Milan, Italy) according to method OIV-MA-AS313-02, reducing substances according to the titrimetric method OIV-MA-AS311-01A, total dry extract using hydrostatic balance "Densimat and Alcomat" Gibertini (Milan, Italy) according to method OIV-MA-AS2-03B, color intensity using spectrophotometer "Helios A" Thermo Fisher Scientific (Waltham, MA, USA) according to method OIV-MA-AS2-07B, hue using spectrophotometer "Helios A" Thermo Fisher Scientific (Waltham, MA, USA) according to method OIV-MA-AS2-07B, total phenolic index (TPI, A280) using spectrophotometer "Helios A" Thermo Fisher Scientific (Waltham, MA, USA), total sulfur dioxide using electronic titrator "Quick" Gibertini (Milan, Italy) according to a validated and accredited method by Hellenic Accreditation System (E.SY.D.) with code "O 1172 In house method", free sulfur dioxide using electronic titrator "Quick" Gibertini (Milan, Italy) according to a validated and accredited method by Hellenic Accreditation System (E.SY.D.) with code "O 1171 In house method".

2.6. Determination of Volatile Aroma Compounds

For the determination of volatile compounds of wine aroma, a method from a previous study by the authors using Gas Chromatograph—Mass Spectrometer—Mass Spectrometer [33] was used.

A Quantum XLS Gas Chromatograph coupled to a Triple Quad Mass Spectrometer by Thermo Scientific Inc. (Waltham, MA, USA) was used. Two microliters of the final extract were injected in splitless mode. The column was TR-Pesticide II (30 m × 0.25 mm ID, 0.25 µm film thickness) by Thermo Scientific Inc. (Waltham, MA, USA); the carrier gas was helium (constant flow 1.0 mL/min); injection inlet temperature was 210 °C; source temperature was 200 °C; transfer line temperature was 250 °C; emission current was 50 µA; experiment type was SRM (selective reaction monitoring); collision gas pressure was 1.5 mTorr; Q1 and Q3 peak width were 0.70; cycle time was 0.500 (s); solvent delay was 7 min; the oven's temperature program was the following: an initial temperature of 40 °C was held for 5 min, increased by 7 °C/min to 170 °C, then increased by 40 °C/min to 290 °C and held for 3 min.

The compounds were identified by their retention times and at least two pairs of fragments. The quantitation was made in selective reaction monitoring (SRM) scan mode, using the fragment's mass transitions with the higher intensity. The detailed MRM (multiple reaction monitoring) parameters, the retention times and the quantification's mass transitions selected for each compound were described in a previous study of authors [33].

2.7. Sensory Analysis

The sensory analysis was conducted by ten trained assessors for the specific method. The method and the training process of the panel were described in a previous study by the authors [34]. For the present study, the parameters smoky aroma and aroma complexity were added to the method that had already been developed.

To train the assessors, a mixture of guaiacol and 4-ethylguaiacol was used for the smoky aroma. For the aroma complexity, a blend of substances was employed, including those contributing to fruity and barrel aromas, specifically, ethyl caproate, ethyl caprylate, isoamyl acetate, vanillin, and whiskey lactone.

To assess these parameters, a model wine (hydroalcoholic solution, with ethanol 12% *v*/*v*, and tartaric acid 5 g/L) was spiked appropriately to create solutions with concentrations approximately 10×, 20×, and 50× their aroma thresholds [37–39]. Specifically, three spiked wine solutions were prepared for the smoky aroma (S1, S2, S3) with the following final concentrations: S1: guaiacol 100 μg/L, 4-ethylguaiacol 300 μg/L; S2: guaiacol 200 μg/L, 4-ethylguaiacol 600 μg/L; S3: guaiacol 500 μg/L, 4-ethylguaiacol 1000 μg/L. Similarly, for aroma complexity, three spiked wine solutions (C1, C2, C3) were created with the following concentrations: C1: ethyl caproate 100 μg/L, ethyl caprylate 50 μg/L, isoamyl acetate 300 μg/L, vanillin 500 μg/L, whiskey lactone 500 μg/L; C2: ethyl caproate 300 μg/L, ethyl caprylate 100 μg/L, isoamyl acetate 600 μg/L, vanillin 800 μg/L, whiskey lactone 800 μg/L; C3: ethyl caproate 700 μg/L, ethyl caprylate 250 μg/L, isoamyl acetate 1500 μg/L, vanillin 1500 μg/L, whiskey lactone 1500 μg/L.

The assessors evaluated these parameters using a continuous 0–10 scale with a 10 cm width. Each assessor recorded their results on a hard copy profile sheet. The data were subsequently read electronically using the specialized software Fizz Calculations, version 2.47B (Biosystemes, Couternon, France).

2.8. Data Analysis

Analyses were conducted on four samples per batch, since the control and each batch of oak chips addition was divided into two sub-batches and each sub-batch was analyzed in duplicate. Subsequently, the average of the four samples for each parameter and each batch was calculated.

Additionally, principal component analysis (PCA) was performed using JMP software 18.0.1. version (SAS, Campus Drive Cary, NC, USA). The purpose was to explore the distinctiveness of certain batches within the entire dataset and identify specific characteristics that might differentiate the parameters. For the PCA, results from both the organoleptic analysis and the determination of volatile compounds of wine's aroma using GC-MS/MS were utilized.

Furthermore, the data underwent one-way analysis of variance (ANOVA) for all the samples and all the parameters analyzed, including the volatile compounds and the sensory attributes. ANOVA was performed using JMP software version 18.0.1 (SAS, Campus Drive, Cary, NC, USA).

3. Results

3.1. Physicochemical Analyses

The physicochemical analysis of the control wine yielded the following results as shown in Table 1. It is important to note that the reported values represent the average of two measurements.

Table 1. Results of the physicochemical analysis for the control wine.

Parameter	Value	Unit
Alcoholic strength by volume	13.73	%*v*/*v*
Density at 20 °C	0.9907	g/mL
pH	3.48	
Total acidity	6.15	g tartaric acid/L
Volatile acidity	0.15	g acetic acid/L
Reducing substances	1.9	g/L
Total dry extract	26.7	g/L
Color intensity	7.78	A
Hue	0.58	
Phenolic’s index	51	A
Total sulfur dioxide	56	mg/L
Free sulfur dioxide	16	mg/L

3.2. Volatile Compounds of Wine Aroma Analysis

Samples were analyzed for thirty-nine aromatic volatile compounds, as shown in Table A1 in the Appendix A. The results are shown only for the compounds whose concentrations were determined to be above the reference limit of the method. The method reference limits for each substance have been previously published in a study by the authors [33]. Volatile compounds originating from the contact of oak chips with wine and determined at concentrations higher than the method’s limit of quantification are shown in Table 2. Vanillin was the only aroma volatile derived from oak chips that was determined at concentrations above the method’s limit of quantification in all samples except the control. More specifically, vanillin was determined at concentrations from 9 μg/L to 86 μg/L and an average of 24 μg/L at 30 days, from 25 μg/L to 278 μg/L and an average of 88 μg/L at 60 days, and from 56 μg/L to 555 μg/L and a mean of 225 μg/L at 90 days.

The compounds responsible for the smoky aroma of the wine 4-ethylguaiacol and guaiacol appear only in the medium toasted and heavy toasted samples. More specifically, 4-ethylguaiacol was determined at a concentration above the limit of quantification only in the heavy toasted 2 g/L, heavy toasted 4 g/l samples at 60 days and in all heavy toasted samples at 90 days. Guaiacol was determined above the limit of quantification in medium toasted 4 g/L, heavy toasted 1 g/L, heavy toasted 2 g/L, heavy toasted 4 g/L at both 30 days, 60 and 90 days, while in medium toasted 2 g/L only at 60 and 90 days.

The results of 2-phenylethanol and acetates, whose concentrations were determined at concentrations above the method’s limit of quantification are shown in Table 3.

The results of the remaining esters, whose concentrations were determined at concentrations above the method’s limit of quantification are shown in Table 4.

Table 2. Concentrations of volatile compounds resulting from the contact with oak chips at 30, 60, and 90 days.

	Acetovanillone (μg/L)			**Ethyl-guaiacol (μg/L)**			**Guaiacol (μg/L)**			**Vanillin (μg/L)**			***trans*-Whiskey Lactone (μg/L)**			***cis*-Whiskey Lactone (μg/L)**		
	30 Days	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**
Control	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
LT_1g	ND	27 ± 10	86 ± 27	ND	ND	ND	ND	ND	ND	11 ± 1	27 ± 10	82 ± 29	ND	ND	ND	19 ± 2	24 ± 3	27 ± 6
LT_2g	12 ± 2	45 ± 2	146 ± 69	ND	ND	ND	ND	ND	ND	13 ± 1	44 ± 3	142 ± 76	ND	20 ± 12	19 ± 7	38 ± 8	62 ± 6	57 ± 16
LT_4g	21 ± 10	73 ± 28	246 ± 96	ND	ND	ND	ND	ND	ND	23 ± 7	70 ± 24	242 ± 117	21 ± 13	42 ± 23	41 ± 12	80 ± 4	117 ± 29	122 ± 11
MT_1g	13 ± 3	28 ± 8	148 ± 5	ND	ND	ND	ND	ND	ND	15 ± 4	25 ± 8	139 ± 13	ND	ND	22 ± 1	13 ± 4	19 ± 7	22 ± 4
MT_2g	22 ± 1	132 ± 28	296 ± 8	ND	ND	ND	ND	10 ± 0	10 ± 2	25 ± 1	126 ± 35	278 ± 26	ND	22 ± 3	33 ± 1	29 ± 4	46 ± 10	44 ± 10
MT_4g	86 ± 20	289 ± 71	600 ± 34	ND	ND	ND	14 ± 2	17 ± 1	20 ± 5	86 ± 14	278 ± 96	550 ± 23	24 ± 7	40 ± 9	52 ± 20	57 ± 8	72 ± 15	85 ± 7
HT_1g	ND	33 ± 5	108 ± 23	ND	ND	5 ± 1	10 ± 0	15 ± 3	15 ± 1	9 ± 2	31 ± 2	100 ± 30	ND	ND	ND	ND	ND	ND
HT_2g	ND	50 ± 17	217 ± 50	ND	7 ± 2	11 ± 1	21 ± 2	27 ± 4	29 ± 2	14 ± 2	49 ± 19	211 ± 68	ND	ND	ND	7 ± 0.4	6 ± 2	11 ± 0.1
HT_4g	21 ± 2	154 ± 43	325 ± 21	ND	15 ± 4	20 ± 3	32 ± 3	48 ± 8	52 ± 2	23 ± 1	143 ± 53	310 ± 52	ND	ND	ND	6 ± 2	15 ± 4	20 ± 3

ND: Not Detected.

Table 3. Concentrations of 2-phenylethanol and acetates during 30, 60, and 90 days of wine contact with oak chips.

	2-Phenylethanol (μg/L)			**2-Phenylethyl-acetate (μg/L)**			**Isoamyl-acetate (μg/L)**			**Isobutyl-acetate (μg/L)**		
	30 Days	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**
Control	40,995 ± 701	43,589 ± 4841	35,453 ± 733	63 ± 3	68 ± 3	66 ± 6	1188 ± 172	1014 ± 100	948 ± 108	69 ± 13	62 ± 10	66 ± 16
LT_1g	40,161 ± 1390	42,612 ± 4912	39,249 ± 1558	65 ± 5	69 ± 4	72 ± 9	1156 ± 108	1019 ± 93	980 ± 88	86 ± 5	75 ± 4	57 ± 7
LT_2g	38,844 ± 1055	40,146 ± 625	40,190 ± 3076	59 ± 2	73 ± 3	69 ± 5	1367 ± 100	1139 ± 69	1028 ± 110	80 ± 8	70 ± 6	49 ± 18
LT_4g	39,979 ± 1283	42,804 ± 3676	38,847 ± 2570	62 ± 8	72 ± 2	67 ± 1	1269 ± 76	1130 ± 119	1035 ± 29	78 ± 5	72 ± 5	58 ± 5
MT_1g	37,357 ± 1526	39,779 ± 8604	39,405 ± 1815	70 ± 4	73 ± 2	68 ± 3	1256 ± 49	1091 ± 47	1041 ± 75	80 ± 5	73 ± 2	62 ± 8
MT_2g	38,968 ± 1525	41,140 ± 2616	38,432 ± 1305	68 ± 3	69 ± 2	65 ± 6	1365 ± 31	1147 ± 52	1009 ± 96	78 ± 8	70 ± 6	60 ± 4
MT_4g	38,129 ± 1641	36,062 ± 5340	38,573 ± 527	70 ± 6	67 ± 2	67 ± 3	1254 ± 56	1001 ± 42	960 ± 9	85 ± 9	69 ± 7	60 ± 6
HT_1g	37,698 ± 1387	42,281 ± 5985	36,877 ± 1913	71 ± 4	69 ± 8	65 ± 4	1303 ± 73	1110 ± 59	1006 ± 130	78 ± 7	69 ± 6	65 ± 10
HT_2g	39,471 ± 5611	40,409 ± 5212	38,650 ± 1349	68 ± 2	67 ± 6	67 ± 5	1189 ± 57	1037 ± 40	1009 ± 44	84 ± 7	72 ± 5	65 ± 3
HT_4g	32,599 ± 1558	35,313 ± 3267	34,954 ± 913	66 ± 1	61 ± 3	57 ± 8	1278 ± 116	1056 ± 106	944 ± 46	83 ± 8	72 ± 7	63 ± 4

Table 4. Concentrations of esters during 30, 60, and 90 days of wine contact with oak chips.

	Ethyl-3-hydroxybutyrate (μg/L)			**Ethyl-butyrate (μg/L)**			**Ethyl-caproate (μg/L)**			**Ethyl-caprylate (μg/L)**			**Ethyl-isobutyrate (μg/L)**		
	30 Days	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**
Control	972 ± 73	813 ± 57	783 ± 59	255 ± 10	261 ± 19	249 ± 21	68 ± 8	56 ± 3	52 ± 3	67 ± 3	71 ± 6	64 ± 6	64 ± 4	61 ± 4	62 ± 5
LT_1g	967 ± 30	801 ± 81	810 ± 32	260 ± 19	248 ± 14	230 ± 8	66 ± 8	58 ± 4	57 ± 2	71 ± 11	68 ± 7	67 ± 6	70 ± 6	65 ± 3	65 ± 5
LT_2g	947 ± 32	787 ± 20	844 ± 85	266 ± 3	247 ± 23	225 ± 16	69 ± 11	60 ± 4	59 ± 4	77 ± 10	76 ± 3	73 ± 7	64 ± 5	61 ± 4	64 ± 4
LT_4g	983 ± 48	1041 ± 351	827 ± 18	262 ± 15	267 ± 17	232 ± 5	75 ± 16	69 ± 7	61 ± 2	82 ± 14	74 ± 6	67 ± 3	71 ± 8	68 ± 7	68 ± 4
MT_1g	919 ± 48	1707 ± 1516	884 ± 45	257 ± 22	252 ± 19	260 ± 46	81 ± 9	70 ± 1	62 ± 3	83 ± 6	77 ± 8	72 ± 3	71 ± 8	70 ± 3	73 ± 4
MT_2g	959 ± 37	817 ± 38	854 ± 34	236 ± 6	260 ± 9	235 ± 7	81 ± 8	66 ± 4	56 ± 3	81 ± 8	80 ± 4	69 ± 3	61 ± 4	60 ± 5	68 ± 5
MT_4g	906 ± 26	802 ± 82	839 ± 42	263 ± 20	253 ± 3	234 ± 14	71 ± 13	63 ± 8	57 ± 2	78 ± 9	73 ± 5	70 ± 6	70 ± 7	66 ± 3	66 ± 5
HT_1g	936 ± 26	795 ± 38	831 ± 75	260 ± 17	256 ± 13	230 ± 16	75 ± 10	62 ± 2	55 ± 7	82 ± 9	78 ± 4	71 ± 4	75 ± 9	68 ± 6	70 ± 5
HT_2g	964 ± 105	731 ± 44	848 ± 24	257 ± 10	255 ± 10	242 ± 9	68 ± 4	55 ± 5	54 ± 2	63 ± 6	68 ± 5	63 ± 3	70 ± 5	65 ± 1	68 ± 6
HT_4g	843 ± 41	684 ± 21	763 ± 43	265 ± 15	244 ± 8	219 ± 10	63 ± 4	57 ± 2	52 ± 3	68 ± 4	67 ± 4	63 ± 2	69 ± 5	65 ± 4	64 ± 2

3.3. Odor Active Values (OAV)

In order to enhance the interpretation of the results and their correlation with the organoleptic analysis, the Odor Active Value (OAV) was calculated for each substance. This calculation was performed for substances that were determined at a concentration exceeding the method's limit of quantification. The OAV is derived using the aroma detection threshold. This threshold is defined as the minimum value of a sensory stimulus that can be perceived [40]. OAV is calculated as

$$\mathrm{OAV} = \mathrm{C}/\mathrm{T}$$

where C is the concentration of the compound and T is the detection threshold of that compound [41].

For the computations, the detection thresholds documented in other studies were utilized. In these specific studies, the aroma perceived by the panel for each substance is also described [37–39,42–46].

The "OAV Oak Compounds" was computed by aggregating the OAVs of the compounds listed in Table 2. Similarly, the "OAV Fruity" was determined by summing up the OAVs of the compounds found in Tables 3 and 4. The results of the OAV calculations and the percentage of "OAV Oak Compounds" for each sample are displayed in Table 5.

Table 5. OAV Oak Compounds, OAV Fruity and percentage of OAV Oak Compounds.

	OAV Fruity			**OAV Oak Compounds**			**%OAV Oak Compounds**		
	30 Days	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**
Control	75.2 ± 5	69.5 ± 2.4	65.1 ± 3	0 ± 0	0 ± 0	0 ± 0	0.0%	0.0%	0.0%
LT_1g	75.3 ± 4.4	68.8 ± 4.1	66.4 ± 3.2	0.1 ± 0.01	0.2 ± 0.06	0.5 ± 0.17	0.1%	0.3%	0.8%
LT_2g	83.9 ± 5.6	74.3 ± 2.5	69 ± 3.7	0.1 ± 0.01	0.6 ± 0.17	1.2 ± 0.54	0.2%	0.8%	1.7%
LT_4g	82.3 ± 6.7	75.7 ± 6.2	68.7 ± 1.4	0.6 ± 0.24	1.2 ± 0.5	2.2 ± 0.84	0.7%	1.6%	3.1%
MT_1g	82.2 ± 4.2	74.5 ± 2.9	71.7 ± 3.1	0.1 ± 0.02	0.2 ± 0.05	1 ± 0.11	0.1%	0.2%	1.4%
MT_2g	83.8 ± 3.2	76.3 ± 2.2	68 ± 3.3	0.2 ± 0.01	2 ± 0.39	3 ± 0.33	0.2%	2.5%	4.2%
MT_4g	80.7 ± 4	70 ± 1.8	66.5 ± 1.2	2.4 ± 0.45	4.2 ± 0.81	6.3 ± 0.76	2.9%	5.7%	8.7%
HT_1g	83.6 ± 4.3	74.8 ± 2.4	68.1 ± 6.4	1.1 ± 0.05	1.7 ± 0.32	2.3 ± 0.07	1.3%	2.3%	3.3%
HT_2g	74.9 ± 1.6	69.6 ± 2.6	67 ± 1.5	2.3 ± 0.18	3.4 ± 0.37	4.7 ± 0.45	2.9%	4.6%	6.5%
HT_4g	78.8 ± 3.2	69.4 ± 3.2	63.3 ± 1.4	3.5 ± 0.29	6.4 ± 0.63	7.9 ± 0.11	4.3%	8.5%	11.2%

When comparing the OAV Oak Compounds, the heavy toasted 4 g/L sample had the highest values starting from 30 days, with a significant difference from the rest of the samples. This was followed by the medium toasted 4 g/L sample and then the heavy toasted 2 g/L sample. The latter two samples had similar values at 30 days, but the difference in their values was increasing at 60 and 90 days, as illustrated in Table 5.

Almost all samples exhibited a value equal to or greater than the control for OAV Fruity, with very few exceptions: at 30 days the high toasted sample at 2 g/L, at 60 days the low toasted sample at 2 g/L, and at 90 days the high toasted sample at 4 g/L. This is depicted in Table 5.

3.4. Sensory Analysis

The results of the sensory analysis, which include olfactory parameters such as barrel aroma, smoky aroma, aroma complexity, and fruity aroma, along with the flavor intensity for each batch of added oak chips and the control, are presented for the durations of 30, 60, and 90 days in Table 6.

Correspondingly, the results of the sensory analysis for the taste attributes are presented in Table 7.

Table 6. Results of sensory analysis for the attributes barrel aroma, smoky aroma, aroma complexity, fruity aroma, flavor intensity.

	Barrel Aroma			**Smoky Aroma**			**Aroma Complexity**			**Fruity Aroma**			**Flavor Intensity**		
	30 Days	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**
Control	0 ± 0	0 ± 0	0 ± 0	0 ± 0	0 ± 0	0 ± 0	1.3 ± 0.1	1.5 ± 0.1	2.6 ± 0.2	5.8 ± 0.3	6.4 ± 0.1	6.5 ± 0.0	4.4 ± 0.1	4.5 ± 0.1	4.4 ± 0.1
LT_1g	0.8 ± 0.1	1 ± 0.1	1.3 ± 0.1	0.2 ± 0	0.4 ± 0.1	0.7 ± 0.1	2.3 ± 0.1	2.6 ± 0.1	3.4 ± 0.1	6 ± 0.1	6.2 ± 0.1	6.3 ± 0.1	4.5 ± 0.2	4.5 ± 0.1	4.4 ± 0.2
LT_2g	1 ± 0.1	1.3 ± 0.1	1.9 ± 0.1	0.5 ± 0	0.6 ± 0.1	0.9 ± 0.2	2.3 ± 0.1	2.8 ± 0.2	4.3 ± 0.1	5.7 ± 0.3	6 ± 0.2	6.1 ± 0.2	4.5 ± 0.3	4.5 ± 0.2	4.6 ± 0.1
LT_4g	1.5 ± 0.2	1.9 ± 0.2	2.8 ± 0.4	0.6 ± 0.1	0.8 ± 0.1	1.4 ± 0.4	2.7 ± 0.1	3.3 ± 0.4	4.7 ± 0.3	6.1 ± 0.2	6.1 ± 0.1	6.2 ± 0.2	4.3 ± 0.3	4.5 ± 0	4.6 ± 0.1
MT_1g	1 ± 0.1	1.1 ± 0.1	2.9 ± 0.1	0.5 ± 0.1	0.7 ± 0.1	1.4 ± 0.1	2.4 ± 0.1	3.2 ± 0.1	4.5 ± 0.3	6.2 ± 0.1	6.2 ± 0.2	6.4 ± 0.2	4.4 ± 0.2	4.7 ± 0.2	4.8 ± 0.2
MT_2g	1.2 ± 0.2	1.5 ± 0.1	3.6 ± 0.2	0.5 ± 0.1	0.9 ± 0.1	2.6 ± 0.2	2.7 ± 0.1	3.3 ± 0.1	4.9 ± 0.2	5.9 ± 0.5	6.1 ± 0.2	6.1 ± 0.1	4.3 ± 0.1	4.8 ± 0.2	4.7 ± 0.1
MT_4g	1.9 ± 0.3	2.1 ± 0.2	4.3 ± 0.1	0.9 ± 0.1	1.3 ± 0.2	2.6 ± 0.3	2.9 ± 0.1	3.6 ± 0.1	5.2 ± 0.2	5.8 ± 0.3	6.1 ± 0.1	6.1 ± 0.3	4.3 ± 0.2	4.8 ± 0.1	4.5 ± 0.1
HT_1g	0.9 ± 0.1	1.1 ± 0.1	2.5 ± 0.2	0.8 ± 0.1	1 ± 0.1	2.1 ± 0.4	2.4 ± 0.1	3.6 ± 0.2	4.8 ± 0.4	5.8 ± 0.3	6.2 ± 0.2	6.1 ± 0.2	4.1 ± 0.1	4.6 ± 0.1	4.3 ± 0.2
HT_2g	1.5 ± 0.1	1.7 ± 0.2	3.1 ± 0.5	1 ± 0.1	1.4 ± 0.1	2.7 ± 0.1	2.7 ± 0.2	3.8 ± 0.5	5.4 ± 0.2	5.7 ± 0.3	6.3 ± 0.2	6.2 ± 0.3	4.3 ± 0.2	4.4 ± 0.1	4.8 ± 0.1
HT_4g	1.5 ± 0.1	1.7 ± 0.3	3.7 ± 0.7	1.3 ± 0.1	1.8 ± 0.1	2.7 ± 0.1	3.3 ± 0.3	4.3 ± 0.3	6.1 ± 0	5.8 ± 0.3	6.3 ± 0.1	6.2 ± 0.2	4.4 ± 0.2	4.5 ± 0.1	5.1 ± 0.1

Table 7. Results of sensory analysis for the taste attributes sour, sweet, bitter, astringent and aftertaste.

	Sour			**Sweet**			**Bitter**			**Astringent**			**Aftertaste**		
	30 Days	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**	**30 Days**	**60 Days**	**90 Days**
Control	4.4 ± 0.1	4.9 ± 0.1	4.6 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.5 ± 0.1	0.5 ± 0.1	0.5 ± 0	2.9 ± 0.8	2.3 ± 0.2	3 ± 0.26	4.3 ± 0.2	4.1 ± 0.3	4.4 ± 0.1
LT_1g	4.4 ± 0	4.6 ± 0.1	4.7 ± 0.1	0.8 ± 0.2	0.8 ± 0.1	0.8 ± 0.1	0.5 ± 0.2	0.5 ± 0	0.6 ± 0.1	2.8 ± 0.1	2.6 ± 0.1	2.9 ± 0.2	4.2 ± 0.1	4.5 ± 0.2	4.3 ± 0.1
LT_2g	4.4 ± 0.1	4.6 ± 0.2	4.7 ± 0.1	0.8 ± 0.1	1 ± 0.1	1 ± 0.1	0.6 ± 0.1	0.6 ± 0.1	0.6 ± 0.1	2.9 ± 0.2	2.9 ± 0.2	3.4 ± 0.2	4.1 ± 0.2	4.3 ± 0.1	4.5 ± 0.2
LT_4g	4.3 ± 0.1	4.3 ± 0.1	4.6 ± 0.1	1.2 ± 0.1	0.8 ± 0.1	0.8 ± 0.1	0.5 ± 0.1	0.5 ± 0	0.5 ± 0	3 ± 0.1	2.7 ± 0.1	3.4 ± 0.1	4.2 ± 0.2	4.5 ± 0.1	4.7 ± 0.2
MT_1g	4.3 ± 0.1	4.5 ± 0.1	4.5 ± 0.1	1 ± 0.1	0.7 ± 0.1	1 ± 0.1	0.4 ± 0.1	0.5 ± 0.1	0.6 ± 0.1	2.8 ± 0.2	3 ± 0.2	3.3 ± 0.1	4.2 ± 0.1	4.5 ± 0.1	4.6 ± 0.1
MT_2g	4.3 ± 0.1	4.8 ± 0.1	4.6 ± 0.1	0.9 ± 0	0.9 ± 0.1	0.8 ± 0	0.5 ± 0.1	0.5 ± 0.1	0.6 ± 0	2.7 ± 0.2	3 ± 0.1	3.2 ± 0.2	4 ± 0.1	4.4 ± 0.1	4.5 ± 0.1
MT_4g	4.2 ± 0.2	4.7 ± 0.2	4.6 ± 0.1	1.1 ± 0.1	1 ± 0.1	1 ± 0.1	0.6 ± 0.1	0.6 ± 0.1	0.8 ± 0.1	2.8 ± 0.3	3.2 ± 0.1	3.7 ± 0.4	4.1 ± 0.2	4.3 ± 0.1	4.5 ± 0.1
HT_1g	4.2 ± 0.1	4.4 ± 0.1	4.6 ± 0.1	0.9 ± 0.2	0.9 ± 0.1	0.8 ± 0	0.6 ± 0.1	0.6 ± 0.1	0.6 ± 0.1	2.9 ± 0.3	3.2 ± 0.1	3.7 ± 0.2	4.1 ± 0.1	4.5 ± 0.1	4.5 ± 0.1
HT_2g	4.3 ± 0.1	4.4 ± 0.1	4.6 ± 0.1	1 ± 0.1	0.9 ± 0.1	0.8 ± 0.1	0.5 ± 0.1	0.6 ± 0.1	0.7 ± 0.1	2.8 ± 0.1	3.3 ± 0.1	3.5 ± 0.1	4.2 ± 0.1	4.5 ± 0.1	4.8 ± 0.05
HT_4g	4.3 ± 0.1	4.5 ± 0.2	4.8 ± 0.1	1.1 ± 0.2	0.9 ± 0	0.9 ± 0	0.5 ± 0.1	0.6 ± 0.1	0.8 ± 0.1	2.8 ± 0.1	3.1 ± 0.1	3.5 ± 0.1	4.2 ± 0.1	4.6 ± 0.1	4.9 ± 0.1

Organoleptically, the barrel aroma was perceived more strongly in the medium toasted 4 g/L sample across all three periods of analysis. The second highest value at 30 days was observed in the samples of low toasted 4 g/L, heavy toasted 2 g/L, and heavy toasted 4 g/L. At 60 days, the sample of low toasted 4 g/L stood out, while at 90 days, the sample of heavy toasted 4 g/L took the lead, as depicted in Table 6.

When comparing the samples for the smoky aroma, the heavy toasted 4 g/L sample had the highest value, while the heavy toasted 2 g/L sample had the second highest value at 30 and 60 days. These two particular samples had identical values at 90 days, as shown in Table 6.

All the aforementioned sensory results are further confirmed by the aroma complexity parameter. The sample of heavy toasted 4 g/L had the highest value across all time periods of the study. The second highest value at 30 days was exhibited by the sample of medium toasted 4 g/L, while at 60 and 90 days, the sample of heavy toasted 2 g/L stood out, as illustrated in Table 6.

In terms of gustatory organoleptic parameters, the bitter samples had similar values at 30 and 60 days. However, at 90 days, three samples, specifically medium toasted 4 g/L, high toasted 2 g/L, and high toasted 4 g/L, displayed increased values compared to the rest, as shown in Table 7.

Similarly, in the astringent category, four samples showed increased values at 90 days, namely the medium toasted 4 g/L and all samples of heavy toasted, as depicted in Table 7.

3.5. Data Analysis

For the Principal Component Analysis (PCA), the results of the parameters related to the aroma were utilized, with a specific emphasis on the aroma attributed to the oak chips. More precisely, the organoleptic parameters such as fruity aroma, barrel aroma, smoky aroma, aroma complexity, flavor intensity, and the compounds determined using GC-MS/MS were included. These compounds encompass acetovanillone, 4-ethylguaiacol, guaiacol, vanillin, *trans*-whiskey lactone, *cis*-whiskey lactone, as well as the sum of Odor Active Values for Fruity (OAV Fruity) and Odor Active Values for Oak Compounds (OAV Oak Compounds).

The results of the PCA are depicted in Figures 1 and 2 below.

The eigenvectors suggest that Principal Component 1 is dependent on OAV Oak compounds, smoky aroma, aroma complexity, acetovanillone, and vanillin. Conversely, Principal Component 2 is dependent on *trans*-Whiskey lactone, *cis*-Whiskey lactone, guaiacol, and 4-ethylguaiacol. The data for the eigenvectors are presented in Table 8.

Table 8. The eigenvectors for PC1 and PC2.

	Prin1	**Prin2**
OAV Esters	−0.21661	0.04312
OAV "Oak" Compounds	0.36676	−0.16566
Fruity Aroma	0.07324	0.09631
Barrel Aroma	0.344	0.12663
Smoky Aroma	0.35498	−0.09741
Aroma Complexity	0.36122	−0.01598
Flavor Intensity	0.1674	0.14881
Acetovanillone	0.33064	0.11128
4-Ethylguaiacol	0.2499	−0.40741
Guaiacol	0.27982	−0.39231
Vanillin	0.34739	0.19066
trans-Whiskey lactone	0.16482	0.50871
cis-Whiskey lactone	0.1115	0.53487

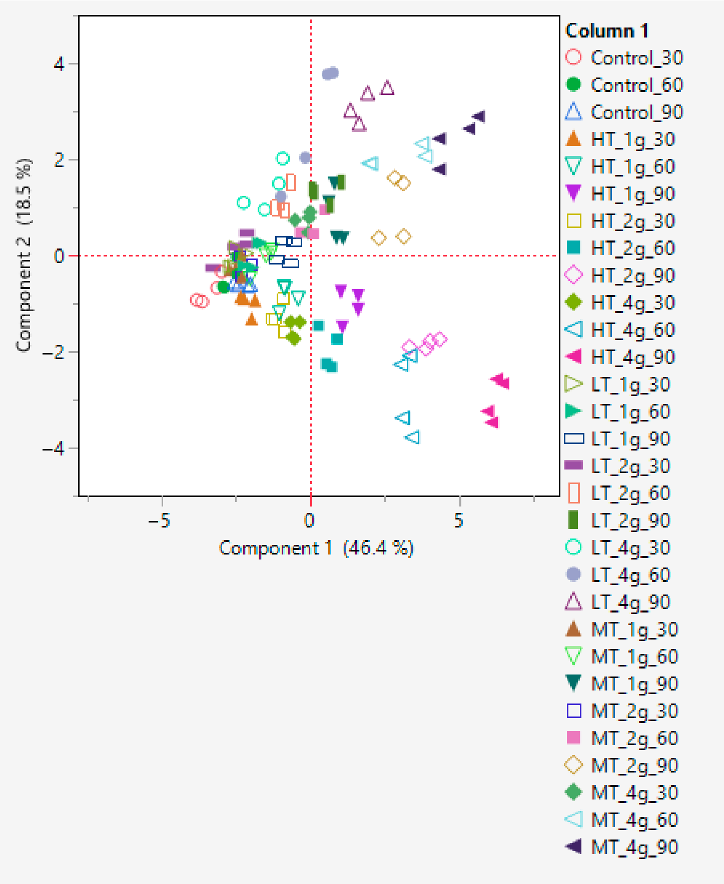

Figure 1. PCA for the sensory attributes fruity aroma, barrel aroma, smoky aroma, aroma complexity, flavor intensity, the compounds acetovanillone, 4-ethylguaiacol, guaiacol, vanillin, *trans*-whiskey lactone, *cis*-whiskey lactone, the OAV Fruity and OAV Oak Compounds in score plot form.

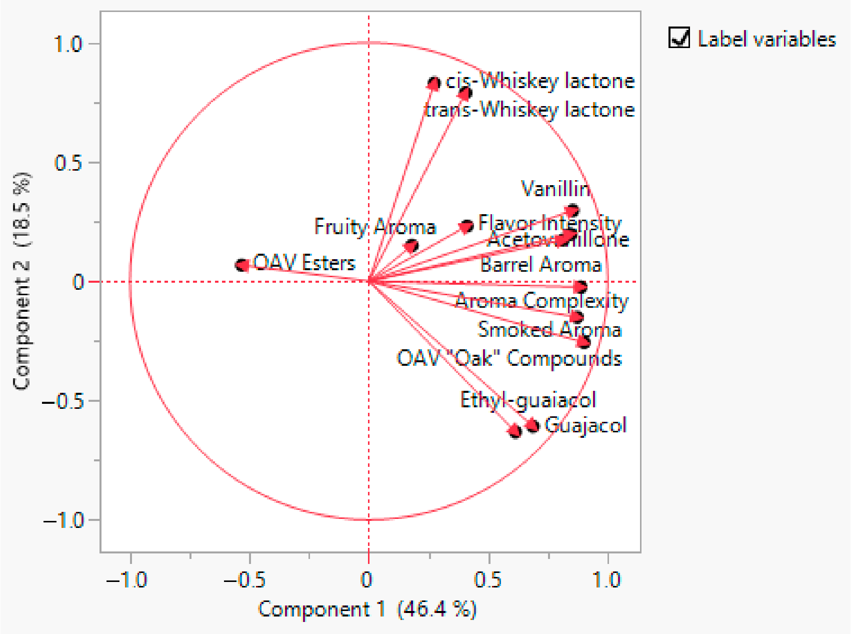

Figure 2. PCA for the sensory attributes fruity aroma, barrel aroma, smoky aroma, aroma complexity, flavor intensity, the compounds acetovanillone, 4-ethylguaiacol, guaiacol, vanillin, *trans*-whiskey lactone, *cis*-whiskey lactone, the OAV Fruity and OAV Oak Compounds in loading plot form.

The one-way analysis of variance (ANOVA) conducted on the analyzed data for all samples revealed that all parameters exhibited a p value less than or equal to 0.05 ($p \leq 0.05$).

4. Discussion

From the current results, it is evident that for OAV Oak Compounds, the dosage is the most significant criterion for increased values, with a dose of 4 g/L leading to a greater extraction of oak-related compounds. A secondary criterion is the toasting intensity of the oak chips, where heavy toasted leads to higher extraction than medium toasted. The OAV Oak Compounds increases and reaches its maximum value in 90 days. These findings align with research by B. Fernández de Simón et al., which suggests that the greatest evolution of volatile compounds, when wines are aged with chips, occurs between 60 and 90 days [19].

This particular study's finding, which shows that most samples exhibited OAV Fruity higher than the control value, aligns with other research but is not directly explainable. More specifically, Antonietta Baiano et al. studied the behavior of must with chips and concluded that the production of esters and acetates during fermentation was higher in the must with chips than in the control [26]. Similarly, D. I. Stegarus et al. studied the influence of oak chips on white wine and concluded that most of the esters had increased concentrations in wines aged with oak chips compared to the control wine [16].

The results of the present research, in relation to the sensory attributes of bitter and astringent, align with previous studies. For instance, Tao et al. stated that wines became more astringent and bitter when heavy-toasted oak chips were used [23], and a study conducted by Koussissi et al. on Agiorgitiko, where oak chips were added during fermentation, concluded that medium and heavy toasted chips made the wines more bitter and astringent [31].

Upon examining the results of GC-MS/MS determinations, it was found that the "key" compounds related to oak are primarily vanillin and, to a lesser extent, the compounds *cis*-whiskey lactone, *trans*-whiskey lactone, guaiacol, and 4-ethylguaiacol. This finding is consistent with a previous study by A.B. Bautista-Ortín et al., who studied the aging of wine with oak chips [17], and also with a study by P. Delgado de la Torre et al. [20].

A deeper analysis of the results for these "key" compounds reveals that *trans*-whiskey lactone appears to be extracted in the first 30 days in medium toasted 4 g/L samples and in all heavy toasted samples, then remains at the same levels. In contrast, the *cis*-whiskey lactone is extracted in the first 30 days, with its concentration increasing at 60 days and remaining at these levels at 90 days. This pattern aligns partially with the conclusion of P. Rubio-Bretón et al., who found that in wines treated with oak chips, the maximum extraction of the two isomers of whiskey lactone occurred during the first month of contact with the wood [27]. In our study, this observation applies to *trans*-whiskey lactone, while *cis*-whiskey lactone is mainly extracted between 30 and 60 days. Both isomers reach their maximum concentration at 90 days, as reported in an earlier study by A.B. Bautista-Ortin et al. [17], where it was also observed that *trans*-whiskey lactone was found in lower concentrations than *cis*-whiskey lactone, a finding that is consistent with the present study.

Vanillin begins its extraction process from 30 days, exhibits a noticeable increase in its concentration at 60 days, and demonstrates a significant surge in concentration at 90 days. There are diverse findings regarding the kinetics of vanillin extraction in wines aged with oak chips. For instance, P. Rubio-Bretón et al. discovered that the maximum concentration of vanillin is reached during the first month [27], while A.B. Bautista-Ortín et al. found that the highest quantities of vanillin were detected in wines after 3 months of aging [17], which is consistent with the current study. Acetovanillone exhibits similar extraction behavior to vanillin. This can be explained by their relationship as oxidation products of lignin. In fact, acetovanillone is an intermediate in the formation of vanillin [47].

The compounds responsible for the smoky aroma, guaiacol and 4-ethylguaiacol, are extracted at 60 days in the heavy toasted oak chips and maintain these levels at 90 days. In medium toasted oak chips, the extraction of guaiacol occurs at 60 days and sustains these levels at 90 days, at the dosages of 2 g/L and 4 g/L. The heavy toasted chips retain higher concentrations of the two compounds than the medium toasted chips when comparing the same time period and respective addition doses, an observation justified as these compounds are formed at high wood toasting temperatures [27].

From the PCA, seven batches appear to be distinct from the rest. Specifically, at 60 days, the medium toasted with the addition of 4 g/L (MT_4g) and the heavy toasted with the addition of 4 g/L (HT_4g) are differentiated. At 90 days, low toasted with the addition of 4 g/L (LT_4g), medium toasted with the addition of 2 g/L (MT_2g), medium toasted with the addition of 4 g/L (MT_4g), heavy toasted with the addition of 2 g/L (HT_2g), and heavy toasted with the addition of 4 g/L (HT_4g) are distinguished. Notably, the low toasted and medium toasted are positioned in the upper right quadrant of the PCA, associated with increased concentrations of vanillin, acetovanillone and whiskey lactone, while the heavy toasted are located in the lower right quadrant of the PCA, associated with the smoky aroma and increased concentrations of guaiacol and 4-ethylguaiacol.

In summary, regarding the seven samples that stand out from the rest during PCA, the heavy toasted 4 g/L is distinguished both at 60 days and at 90 days, as it exhibited the highest values of Oak Compounds due to the extraction of guaiacol and 4-ethylguaiacol, acetovanillone, as well as vanillin. Simultaneously, the extraction of guaiacol and 4-ethylguaiacol led to increased values of the sensory attributes of smoky aroma and aroma complexity, and at 90 days, it also showed increased bitter and astringent values.

The heavy toasted 2 g/L differs from the rest only at 90 days, displaying relatively increased values of OAV Oak Compounds, smoky aroma and aroma complexity, as well as bitter and astringent sensory attributes, albeit at lower values than those in heavy toasted 4 g/L.

Medium toasted 4 g/L stands out from the rest both at 60 days and at 90 days, having a higher value of OAV Oak Compounds and the highest value in the sensory attribute of barrel aroma due to the more significant concentrations of vanillin and acetovanillone compared to the rest of the samples. In terms of gustatory organoleptic parameters, medium toasted 4 g/L showed higher values of bitter and astringent than most samples.

The medium toasted 2 g/L is differentiated from the rest only at 90 days, displaying high values in the organoleptic parameter of barrel aroma and high concentrations of vanillin and acetovanillone, but at lower values than the dose of 4 g/L.

The dose of 4 g/L is distinctive among the rest of the samples at 90 days and in low toasted. This particular sample had the fourth highest concentration of vanillin and acetovanillone, while simultaneously having higher OAV fruity values compared to the control sample.

Comparing the results of the current experiment with a previous study by the authors, where similar analyses were conducted on Nemea PDO sample products [33,34], we can extract useful information. In none of the samples from the current study, where oak chips were added, did we observe in the sensory analysis a value of barrel aroma higher than fruity aroma. In contrast, in the Nemea products, 28% of the samples exhibited higher values of barrel aroma than fruity. In the Nemea products from the previous study, the average OAV Oak Compounds was 4.4. Values higher than this were displayed by heavy toasted 4 g/L at 60 and 90 days, as well as medium toasted 4 g/L, heavy toasted 2 g/L at 90 days. In the barrel aroma sensory attribute, the average for Nemea products was 3.2. Higher values in barrel aroma were shown by the samples at 90 days, medium toast 2 g/L, medium toast 4 g/L and heavy toasted 4 g/L, while heavy toasted 2 g/L showed values very close to the average. From these data and the specific comparison, it can be inferred that the addition of oak chips is a method that generally produces products with a lower wood sensation compared to the traditional barrel aging method.

5. Conclusions

Aging with the use of oak chips significantly enhances the aromatic profile of wines from the Agiorgitiko variety, with nearly all samples aged with oak chips exhibiting higher concentrations of esters, as reflected by the Odor Active Value of esters, compared to the control. The samples aged with heavy toasted chips generally display a higher value of the sensory attribute smoky aroma, associated with the compounds guaiacol and 4-ethylguaiacol, while the medium toasted chips exhibit a higher value of the sensory attribute barrel aroma, primarily related to the compounds vanillin, acetovanillone and whiskey lactone. The organoleptic parameter aroma complexity is mainly linked to the dosage of the addition and less to the degree of toasting, and in the bitter and astringent sensory attributes, some samples were only distinctive among the rest of the samples at 90 days of aging.

In relation to oak-related volatile compounds, vanillin and acetovanillone begin their extraction from 30 days and continue to be extracted until 90 days, *trans*-whiskey lactone appears to be extracted for the first 30 days and then remains at the same levels, and *cis*-whiskey lactone is extracted in the first 30 days, increases at 60 days and remains at these levels at 90 days. Meanwhile, the substances guaiacol and 4-ethylguaiacol are extracted at 60 days in heavy toasted oak chips and remain at these levels at 90 days.

When comparing the results of the wines created during this study with the results of PDO Nemea samples, the high doses (4 g/L) of medium and heavy toasting showed values higher than the average of the commercial samples, both for OAV Oak Compounds and the sensory attribute barrel aroma, at 90 days. From the comparison of the data of the current study with the authors' previous data on commercial PDO Nemea samples, it can be inferred that the aging of Agiorgitiko with oak chips leads to products with less oak sensation, compared to the traditional barrel aging method.

Finally, from the extensive amount of data presented here, oenologists and winemakers who produce wines from the Agiorgitiko variety can select the degree of toasting, the dosage, and the aging time that they deem appropriate for the wine they wish to age, but also with the product they aim to create by making the relevant predictions.

Author Contributions: Conceptualization, I.L.; methodology, I.L.; software, I.L.; validation, I.L.; formal analysis, I.L.; investigation, I.L.; resources, I.L.; data curation, I.L.; writing—original draft preparation, I.L.; writing—review and editing, I.L. and Y.K.; visualization, I.L.; supervision, I.L. and Y.K.; project administration, I.L.; funding acquisition, I.L. All authors have read and agreed to the published version of the manuscript.

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Informed Consent Statement: Informed consent was obtained from all the subjects involved in the study.

Data Availability Statement: The data that support the findings of this study are available upon reasonable request from the authors.

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Conflicts of Interest: The authors declare no conflicts of interest.

Appendix A

Table A1. List of standards, Chemical Abstracts Service Number (CAS Registry Number), molecular formula, relative formula mass (Mr), purity, and company.

a/a	Name	CAS	Molecular Formula	Mr	Purity	Company
1	2-Methoxy-4-methylphenol	93-51-6	$C_8H_{10}O_2$	138.16	99.6%	Sigma Aldrich (St. Louis, MO, USA)
2	2-Methoxy-4-vinylphenol	7786-61-0	$C_9H_{10}O_2$	150.17	99.7%	Sigma Aldrich (St. Louis, MO, USA)
3	2-Phenethyl acetate	103-45-7	$C_{10}H_{12}O_2$	164.2	99.5%	Honeywell Fluka (Charlotte, NC, USA)
4	2-Phenylethanol	60-12-8	$C_8H_{12}O$	122.16	99.3%	Sigma Aldrich (St. Louis, MO, USA)
5	3-(Methylthio)propionaldehyde	3268-49-3	C_4H_8SO	104.17	97.2%	Sigma Aldrich (St. Louis, MO, USA)
6	4-Ethylguaiacol	2785-89-9	$C_9H_{12}O_2$	152.19	98.0%	Sigma Aldrich (St. Louis, MO, USA)
7	4-Ethylphenol	123-07-9	$C_8H_{10}O$	122.17	99.2%	Acros Organics (Geel, Belgium)
8	4-Vinylphenol solution 10 wt.%	2628-17-3	C_8H_8O	120.15	96.0%	Sigma Aldrich (St. Louis, MO, USA)
9	Acetovanillone	498-02-2	$C_9H_{10}O_3$	166.17	98.0%	Sigma Aldrich (St. Louis, MO, USA)
10	ß-Ionone	79-77-6	$C_{13}H_{20}O$	192.3	97.1%	Honeywell Fluka (Charlotte, NC, USA)
11	Benzyl acetate	140-11-4	$C_9H_{10}O_2$	150.17	99.9%	Sigma Aldrich (St. Louis, MO, USA)
12	Citral	5392-40-5	$C_{10}H_{16}O$	152.23	96.0%	Sigma Aldrich (St. Louis, MO, USA)
13	Citronelol	106-22-9	$C_{10}H_{20}O$	156.27	95.0%	Acros Organics (Geel, Belgium)
14	Damascenone natural	23696-85-7	$C_{13}H_{18}O$	190.28	1.1–1.4 wt.%	Sigma Aldrich (St. Louis, MO, USA)
15	Decyl aldehyde	112-31-2	$C_{10}H_{20}O$	156.27	98.5%	Acros Organics (Geel, Belgium)
16	Ethyl 2-methylbutyrate	7452-79-1	$C_7H_{14}O_2$	130.19	99.3%	Acros Organics (Geel, Belgium)
17	Ethyl 3-hydroxybutyrate	5405-41-4	$C_6H_{12}O_3$	132.16	99.6%	Sigma Aldrich (St. Louis, MO, USA)
18	Ethyl butyrate	105-54-4	$C_6H_{12}O_2$	116.16	≥98.0%	Honeywell Fluka (Charlotte, NC, USA)
19	Ethyl caproate	123-66-0	$C_8H_{16}O_2$	144.21	99.7%	Acros Organics (Geel, Belgium)
20	Ethyl caprylate	106-32-1	$C_{10}H_{20}O_2$	172.26	99.2%	Sigma Aldrich (St. Louis, MO, USA)
21	Ethyl cinnamate trans	103-36-6	$C_{11}H_{12}O_2$	176.21	99.7%	Acros Organics (Geel, Belgium)
22	Ethyl decanoate	110-38-3	$C_{12}H_{24}O_2$	200.32	99.7%	Sigma Aldrich (St. Louis, MO, USA)
23	Ethyl dodecanoate	106-33-2	$C_{14}H_{28}O_2$	228.37	99.7%	Sigma Aldrich (St. Louis, MO, USA)
24	Ethyl isobutyrate	97-62-1	$C_6H_{12}O_2$	116.16	99.3%	Honeywell Fluka (Charlotte, NC, USA)
25	Ethyl isovalerate	108-64-5	$C_7H_{14}O_2$	130.18	99.7%	Honeywell Fluka (Charlotte, NC, USA)
26	Ethyl vanillin	121-32-4	$C_9H_{10}O_3$	166.17	97.0%	Acros Organics (Geel, Belgium)
27	Eugenol	97-53-0	$C_{10}H_{12}O_2$	164.2	99.9%	Acros Organics (Geel, Belgium)
28	Geraniol	106-24-1	$C_{10}H_{18}O$	154.25	99.0%	Acros Organics (Geel, Belgium)
29	Guaiacol	90-05-1	$C_7H_8O_2$	124.14	99.5%	Sigma Aldrich (St. Louis, MO, USA)
30	Hexanal	66-25-1	$C_6H_{12}O$	100.16	≥97.5%	Sigma Aldrich (St. Louis, MO, USA)
31	Hexyl acetate	142-92-7	$C_8H_{16}O_2$	144.21	≥98.5%	Honeywell Fluka (Charlotte, NC, USA)
32	Isoamyl acetate	123-92-2	$C_7H_{14}O_2$	130.19	≥99.0%	Acros Organics (Geel, Belgium)
33	Isobutyl acetate	110-19-0	$C_6H_{12}O_2$	116.16	≥98.5%	Honeywell Fluka (Charlotte, NC, USA)
34	Isoeugenol	97-54-1	$C_{10}H_{12}O_2$	164.2	99.3%	Sigma Aldrich (St. Louis, MO, USA)
35	Linalool	78-70-6	$C_{10}H_{18}O$	154.25	98.5%	Acros Organics (Geel, Belgium)
36	Rose Oxide	16409-43-1	$C_{10}H_{18}O$	154.25	99.9%	Honeywell Fluka (Charlotte, NC, USA)
37	Thymol	89-83-8	$C_{10}H_{14}O$	150.22	99.9%	Sigma Aldrich (St. Louis, MO, USA)
38	Vanillin	121-33-5	$C_8H_8O_3$	152.15	99.5%	Acros Organics (Geel, Belgium)
39	Whiskey lactone	39212-23-2	$C_9H_{16}O_2$	156.22	99.4%	Sigma Aldrich (St. Louis, MO, USA)

References

1. Garde-Cerdán, T.; Lorenzo, C.; Carot, J.M.; Esteve, M.D.; Climent, M.D.; Salinas, M.R. Effects of composition, storage time, geographic origin and oak type on the accumulation of some volatile oak compounds and ethylphenols in wines. *Food Chem.* **2010**, *122*, 1076–1082. [CrossRef]
2. Garde-Cerdán, T.; Ancín-Azpilicueta, C. Effect of oak barrel type on the volatile composition of wine: Storage time optimization. *LWT* **2006**, *39*, 199–205. [CrossRef]

3. Pérez-Prieto, L.J.; López-Roca, J.M.; Martínez-Cutillas, A.; Pardo Mínguez, F.; Gómez-Plaza, E. Maturing Wines in Oak Barrels. Effects of Origin, Volume, and Age of the Barrel on the Wine Volatile Composition. *J. Agric. Food Chem.* **2002**, *50*, 3272–3276. [CrossRef] [PubMed]
4. Fernández De Simón, B.; Cadahía, E.; Jalocha, J. Volatile Compounds in a Spanish Red Wine Aged in Barrels Made of Spanish, French, and American Oak Wood. *J. Agric. Food Chem.* **2003**, *51*, 7671–7678. [CrossRef]
5. Pérez-Prieto, L.J.; López-Roca, J.M.; Martínez-Cutillas, A.N.; Pardo-Mínguez, F.; Gómez-Plaza, E. Extraction and Formation Dynamic of Oak-Related Volatile Compounds from Different Volume Barrels to Wine and Their Behavior during Bottle Storage. *J. Agric. Food Chem.* **2003**, *51*, 5444–5449. [CrossRef]
6. Garde-Cerdán, T.; Ancín-Azpilicueta, C. Review of quality factors on wine ageing in oak barrels. *Trends Food Sci. Technol.* **2006**, *17*, 438–447. [CrossRef]
7. Spillman, P.J.; Iland, P.G.; Sefton, M.A. Accumulation of volatile oak compounds in a model wine stored in American and Limousin oak barrels. *Aust. J. Grape Wine Res.* **1998**, *4*, 67–73. [CrossRef]
8. Garde Cerdán, T.; Torrea Goñi, D.; Ancín Azpilicueta, C. Changes in the concentration of volatile oak compounds and esters in red wine stored for 18 months in re-used French oak barrels. *Aust. J. Grape Wine Res.* **2002**, *8*, 140–145. [CrossRef]
9. Spillman, P.J.; Pollnitz, A.P.; Liacopoulos, D.; Skouroumounis, G.K.; Sefton, M.A. Accumulation of Vanillin during Barrel-Aging of White, Red, and Model Wines. *J. Agric. Food Chem.* **1997**, *45*, 2584–2589. [CrossRef]
10. Jarauta, I.; Cacho, J.; Ferreira, V. Concurrent Phenomena Contributing to the Formation of the Aroma of Wine during Aging in Oak Wood: An Analytical Study. *J. Agric. Food Chem.* **2005**, *53*, 4166–4177. [CrossRef]
11. Ferreira, V.; Jarauta, I.; Cacho, J. Physicochemical Model To Interpret the Kinetics of Aroma Extraction during Wine Aging in Wood. Model Limitations Suggest the Necessary Existence of Biochemical Processes. *J. Agric. Food Chem.* **2006**, *54*, 3047–3054. [CrossRef] [PubMed]
12. International Organisation of Vine and Wine. International Code of Oenological Practices. Available online: https://www.oiv.int/public/medias/8630/code-2022-en.pdf (accessed on 31 January 2024).
13. European Union. Council Regulation (EC) No 2165/2005. Available online: https://eur-lex.europa.eu/legal-content/EN/ALL/?uri=CELEX:32005R2165 (accessed on 20 December 2005).
14. European Union. Commission Delegated Regulation (EU) 2019/934. Available online: https://eur-lex.europa.eu/eli/reg_del/2019/934/oj (accessed on 12 March 2019).
15. Laqui-Estaña, J.; López-Solís, R.; Peña-Neira, A.; Medel-Marabolí, M.; Obreque-Slier, E. Wines in contact with oak wood: The impact of the variety (Carménère and Cabernet Sauvignon), format (barrels, chips and staves) and aging time on the phenolic composition. *J. Sci. Food Agric.* **2019**, *99*, 436–448. [CrossRef]
16. Stegăruș, D.I.; Călugăr, A.; Tanase, C.; Muscă, A.; Botoran, O.R.; Manolache, M.; Babeș, A.C.; Bunea, C.; Gál, E.; Bunea, A.; et al. Influence of Oak Chips and Oak Barrel Ageing on Volatile Profile in Chardonnay Wine of Romania. *Appl. Sci.* **2021**, *11*, 3691. [CrossRef]
17. Bautista-Ortín, A.B.; Lencina, A.G.; Cano-López, M.; Pardo-Mínguez, F.; López-Roca, J.M.; Gómez-Plaza, E. The use of oak chips during the ageing of a red wine in stainless steel tanks or used barrels: Effect of the contact time and size of the oak chips on aroma compounds. *Aust. J. Grape Wine Res.* **2008**, *14*, 63–70. [CrossRef]
18. Călugăr, A.; Coldea, T.E.; Pop, C.R.; Pop, T.I.; Babeș, A.C.; Bunea, C.I.; Manolache, M.; Gal, E. Evaluation of Volatile Compounds during Ageing with Oak Chips and Oak Barrel of Muscat Ottonel Wine. *Processes* **2020**, *8*, 1000. [CrossRef]
19. Fernández de Simón, B.; Cadahía, E.; del Álamo, M.; Nevares, I. Effect of size, seasoning and toasting in the volatile compounds in toasted oak wood and in a red wine treated with them. *Anal. Chim. Acta* **2010**, *660*, 211–220. [CrossRef]
20. Delgado de la Torre, P.; Priego-Capote, F.; Luque de Castro, D. Comparative profiling analysis of woody flavouring from vine-shoots and oak chips. *J. Sci. Food Agric.* **2014**, *94*, 504–514. [CrossRef] [PubMed]
21. Fanzone, M.; Catania, A.; Assof, M.; Jofré, V.; Prieto, J.; Gil Quiroga, D.; Lacognata Sottano, J.; Sari, S. Application of Vine-Shoot Chips during Winemaking and Aging of Malbec and Bonarda Wines. *Beverages* **2021**, *7*, 51. [CrossRef]
22. Cejudo-Bastante, M.J.; Hermosín-Gutiérrez, I.; Pérez-Coello, M.S. Micro-oxygenation and oak chip treatments of red wines: Effects on colour-related phenolics, volatile composition and sensory characteristics. Part II: Merlot wines. *Food Chem.* **2011**, *124*, 738–748. [CrossRef]
23. Tao, Y.; García, J.F.; Sun, D.W. Advances in Wine Ageing Technologies for Enhancing Wine Quality and Accelerating Wine Ageing Process. *Crit. Rev. Food Sci. Nutr.* **2013**, *54*, 817–835. [CrossRef]
24. Martínez-Gil, A.M.; del Alamo-Sanza, M.; Gutiérrez-Gamboa, G.; Moreno-Simunovic, Y.; Nevares, I. Volatile composition and sensory characteristics of Carménère wines macerating with Colombian (*Quercus humboldtii*) oak chips compared to wines macerated with American (*Q. alba*) and European (*Q. petraea*) oak chips. *Food Chem.* **2018**, *266*, 90–100. [CrossRef] [PubMed]
25. Tavares, M.; Jordão, A.M.; Ricardo-da-Silva, J.M. Impact of cherry, acacia and oak chips on red wine phenolic parameters and sensory profile. *OENO One* **2017**, *51*, 329. [CrossRef]
26. Baiano, A.; De Gianni, A. Timing of the treatment with oak chips: The case of Nero di Troia wine. *Eur. Food Res. Technol.* **2016**, *242*, 1343–1353. [CrossRef]
27. Rubio-Bretón, P.; Garde-Cerdán, T.; Martínez, J. Use of Oak Fragments during the Aging of Red Wines. Effect on the Phenolic, Aromatic, and Sensory Composition of Wines as a Function of the Contact Time with the Wood. *Beverages* **2018**, *4*, 102. [CrossRef]

28. Gordillo, B.; Baca-Bocanegra, B.; Rodriguez-Pulído, F.J.; González-Miret, M.L.; Estévez, I.G.; Quijada-Morín, N.; Heredia, F.J.; Escribano-Bailón, M.T. Optimisation of an oak chips-grape mix maceration process. Influence of chip dose and maceration time. *Food Chem.* **2016**, *206*, 249–259. [CrossRef] [PubMed]
29. Hernández-Carapia, M.Á.; Verde-Calvo, J.R.; Escalona-Buendía, H.B.; Peña-Álvarez, A. Effect of Maturation with American Oak Chips on the Volatile and Sensory Profile of a Cabernet Sauvignon Rosé Wine and Its Comparison with Commercial Wines. *Beverages* **2023**, *9*, 72. [CrossRef]
30. Baiano, A.; De Gianni, A.; Mentana, A.; Quinto, M.; Centonze, D.; Del Nobile, M.A. Effects of the treatment with oak chips on color-related phenolics, volatile composition, and sensory profile of red wines: The case of Aglianico and Montepulciano. *Eur. Food Res. Technol.* **2016**, *242*, 745–767. [CrossRef]
31. Koussissi, E.; Dourtoglou, V.G.; Ageloussis, G.; Paraskevopoulos, Y.; Dourtoglou, T.; Paterson, A.; Chatzilazarou, A. Influence of toasting of oak chips on red wine maturation from sensory and gas chromatographic headspace analysis. *Food Chem.* **2009**, *114*, 1503–1509. [CrossRef]
32. Kyraleou, M.; Kallithraka, S.; Chira, K.; Tzanakouli, E.; Ligas, I.; Kotseridis, Y. Differentiation of Wines Treated with Wood Chips Based on Their Phenolic Content, Volatile Composition, and Sensory Parameters. *J. Food Sci.* **2015**, *80*, C2701–C2710. [CrossRef]
33. Ligas, I.; Goulioti, E.; Tarantilis, P.; Kotseridis, Y. A New Simple Method for the Determination of Complex Wine Aroma Compounds Using GC-MS/MS—The Case of the Greek Variety "Agiorgitiko". *AppliedChem* **2024**, *4*, 122–139. [CrossRef]
34. Ligas, I.; Kotseridis, Y. Introducing a Standardized Sensory Analysis Method for Wine: A Methodology for the Recruitment, Selection, Training, and Monitoring of Assessors—Implementation on the Greek Variety "Agiorgitiko". *Beverages* **2024**, *10*, 63. [CrossRef]
35. International Organisation of Vine and Wine. Compendium Of International Methods of Wine And Must Analysis. Available online: https://www.oiv.int/standards/compendium-of-international-methods-of-wine-and-must-analysis (accessed on 31 January 2024).
36. Hellenic Accreditation System (ESYD). Scope of Accreditation of the Testing Laboratory CADMION. Available online: https://esyd.gr/cadmion-i-%CE%BB%CE%AF%CE%B3%CE%BA%CE%B1%CF%82-%CF%83%CE%B9%CE%B1-%CE%BF%CE%B5/ (accessed on 18 December 2023).
37. Ferreira, V.; Lopez, R.; Cacho, J.F. Quantitative determination of the odorants of young red wines from different grape varieties. *J. Sci. Food Agric.* **2000**, *80*, 1659–1667. [CrossRef]
38. López, R.; Ortín, N.; Pérez-Trujillo, J.P.; Cacho, J.; Ferreira, V. Impact odorants of different young white wines from the Canary Islands. *J. Sci. Food Agric.* **2003**, *51*, 3419–3425. [CrossRef] [PubMed]
39. Campo, E.; Ferreira, V.; Escudero, A.; Marqués, J.C.; Cacho, J. Quantitative gas chromatography-olfactometry and chemical quantitative study of the aroma of four Madeira wines. *Anal. Chim. Acta* **2006**, *563*, 180–187. [CrossRef]
40. European Accreditation. *Accreditation for Sensory Testing Laboratories. EA-4/09*; European Accreditation: Paris, France, 2003.
41. Antia, G.; Pereira, M.; Fraga, P.; Garcia-Oliveira, M.; Carpena, C.; Jimenez-Lopez, C.; Lourenço-Lopes, L.; Barros, L.; Isabel, C.F.R.; Ferreira, M.A.; et al. Management of Wine Aroma Compounds: Principal Basis and Future Perspectives. In *Chemistry and Biochemistry of Winemaking, Wine Stabilization and Aging*; IntechOpen: London, UK, 2020. [CrossRef]
42. Noguerol-Pato, R.; González-Barreiro, C.; Cancho-Grande, B.; Simal-Gándara, J. Quantitative determination and characterization of the main odourants of Mencía monovarietal red wines. *Food Chem.* **2009**, *117*, 473–484. [CrossRef]
43. Vázquez-Pateiro, I.; Arias-González, U.; Mirás-Avalos, J.M.; Falqué, E. Evolution of the Aroma of Treixadura Wines during Bottle Aging. *Foods* **2020**, *9*, 1419. [CrossRef]
44. De-La-Fuente-Blanco, A.; Sáenz-Navajas, M.P.; Valentin, D.; Ferreira, V. Fourteen ethyl esters of wine can be replaced by simpler ester vectors without compromising quality but at the expense of increasing aroma concentration. *Food Chem.* **2020**, *307*, 125553. [CrossRef]
45. Renault, P.; Coulon, J.; de Revel, G.; Barbe, J.C.; Bely, M. Increase of fruity aroma during mixed *T. delbrueckii*/*S. cerevisiae* wine fermentation is linked to specific esters enhancement. *Int. J. Food Microbiol.* **2015**, *207*, 40–48. [CrossRef]
46. Rocha, S.M.; Rodrigues, F.; Coutinho, P.; Delgadillo, I.; Coimbra, M.A. Volatile composition of Baga red wine: Assessment of the identification of the would-be impact odourants. *Anal. Chim. Acta* **2004**, *513*, 257–262. [CrossRef]
47. Rodrigues Pinto, P.C.; Borges da Silva, E.A.; Rodrigues, A.E. Lignin as Source of Fine Chemicals: Vanillin and Syringaldehyde. In *Biomass Conversion*; Baskar, C., Baskar, S., Dhillon, R., Eds.; Springer: Berlin/Heidelberg, Germany, 2012; pp. 381–420. [CrossRef]