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Aromatic and Sensory Characterization of Maturana Blanca Wines Made with Different Technologies

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Abstract: *Vitis vinífera* L. cv. Maturana Blanca is an autochthonous minor variety recently recovered in the Rioja Qualified Denomination of Origin (D.O.Ca Rioja, Spain) for the production of monovarietal white wines with singular and differentiated characteristics. In this paper, Maturana Blanca wines made with different technologies were analyzed by sensory analysis and aromatic profile by gas chromatography-mass detector. Maturana Blanca wines were characterized by low pH, high acidity, and yellowish tonalities. The compounds that most influenced the aroma of Maturana Blanca wines were those related to fruity (acetates and ethyl esters), floral aromas (2-phenylethanol), and spicy notes (γ-decalactone). These wines were mainly characterized by volatile compounds of fruity aromas of banana and apple. The use of pre-fermentative maceration increased the concentration of ethyl esters and acetates and produced wines with higher odor activity values, indicating a greater aromatic intensity. The aromatic profile of Maturana Blanca wines fermented in oak barrels showed a greater complexity as they were also characterized by the presence of important amounts of furfural, whiskey lactone, and eugenol. The sensory analysis confirmed the results obtained in the aromatic analysis, and described the wines as fresh and balanced in mouth, with notes of acidity and medium to high persistence. These results will contribute to a better knowledge of this white variety.

Keywords: monovarietal white wines; pre-fermentative maceration; oak barrel fermentation; volatile compounds; wine aroma

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1. Introduction

Aromas are one of the main parameters of white wines wine quality. Wine aroma is produced by the combination of different compounds belonging to very heterogeneous chemical groups such as aldehydes, esters, alcohols, terpenes, acids, ketones, etc. The volatile compounds in varietal white wines come not only from the grape variety but also from the yeast used during fermentation along with other winemaking practices [1,2]. The volatile compounds from the grape are determinant in the wine quality and typicity, but most of the aroma components are formed as secondary products during alcoholic fermentation without forgetting that oak provides the wine with different volatile substances [3,4].

White wines are commonly elaborated by direct fermentation of free run juice and press fractions in stainless steel deposits. Mixing of the press fractions can produce a more balanced wine and contribute to higher aroma complexity in the wine. White winemaking with pre-fermentative maceration or *skin contact* is made to favor the release of aromatic compounds, which are mainly located in the skins, and increase aromatic intensity. Hence, several studies point out that this technique significantly affects the volatile characteristics of the wines, providing to the wine's higher aroma complexity [5–7]. When white wines are fermented in barrels, the oak-derived compounds will impact on the olfactory and gustatory perception of the wines, which will be dependent on the type of specie and geographical origin of oak, size and toasting of the barrel, barrel age, time of aging, etc. [8].

New market strategies are emerging in the wine industry to diversify wine production and enhance the particularities of different grape cultivars. In recent years, the Rioja Qualified Denomination of Origin (D.O.Ca Rioja, Spain) has promoted the recovery of local white minority varieties in order to preserve its grapevine biodiversity, and also diversify its wines and attract consumers in times when white wines are trendy. In this sense, Maturana Blanca *Vitis vinifera* L. var. is more and more used by some Rioja wineries to elaborate wines with personality and different from the typical Rioja white wines. However, there are no published studies on the chemical and volatile composition of Maturana Blanca wines, and neither in the different technologies that could affect their sensory quality.

The Maturana Blanca white variety, authorized in 2008 by the D.O.Ca Rioja, is the oldest variety of which there is written knowledge in Rioja, since it was already mentioned in 1622 as "Rivadavia", and was considered one of the main white varieties in La Rioja. It comes from the hybridization between Castelana Blanca (mother) and Savagnin Blanc (father), and therefore it is the sister of other varieties such as Godello and Verdejo [9]. The white Maturana currently accounts for 0.58% of the total white varieties in the D.O.Ca Rioja, with 38.82 ha cultivated in 2019. It is a fairly fertile variety with small bunches and berries. The most notable characteristics of the Maturana Blanca variety are its low pH and high acidity, which compensate for the high degree that this variety can reach. In the organoleptic analysis, it presents a very good evaluation. Maturana Blanca wines have been described as greenish-yellow in color; with fruity aromas of apple, banana, and citrus, as well as herbaceous notes; light, fresh, and balanced in mouth, and with slightly notes of acidity and a soft final bitterness [10]. Maturana Blanca is a variety with high aging capacity, and whose aromatic profile could give surprises in combination with the passage through the wood. However, the chemical and sensory characteristics of a varietal wine may change depending on the winemaking method used.

This work has been carried out with two main objectives: (i) characterize the volatile composition and sensory properties of varietal wines made from Maturana Blanca, and (ii) know the influence of different winemaking techniques on the volatile composition and sensory properties of Maturana Blanca wines. The elaboration has been carried out in the experimental winery of Bodegas Campo Viejo (Grupo Pernod Ricard Bodegas, Logroño, Spain). The techniques used have been conventional winemaking of white wines, conventional winemaking with pre-fermentation maceration, and barrel fermentation. All these elaborations will clearly affect the sensory and volatile characteristics of the resulting white wines.

2. Materials and Methods

2.1. Grapes and Winemaking

Grapes from Maturana Blanca variety were collected during the 2018 vintage from a vineyard of Bodegas Campo Viejo (Grupo Pernod Ricard Bodegas, Logroño, Spain). The vineyard was located in Ordoyo, in the D.O.Ca Rioja. Grapes (3000 kg) were harvested by hand at commercial ripeness and at optimum sanitary state at early morning; they were placed in 10-kg plastic boxes, transported to the Bodegas Campo Viejo winery and immediately processed.

Grapes were firstly carefully selected in a selection table where leaves and undesired clusters were removed. Then, three winemaking procedures were carried out in triplicate: (i) conventional winemaking (CW), (ii) conventional winemaking with pre-fermentative maceration (PM), (iii) fermentation in barrel (BF).

For conventional winemaking (CW), grapes were destemmed, crushed, and sulfited (0.02 g/L). Thereafter, they were directly pressed in a pneumatic press (BucherVaslin XPro 8, Chalonnes-sur-Loire, France) where pectolytic enzymes were added (0.01 g/L, Lafase XL Press, Laffort, Bordeaux, France). The free run juice was quickly clarified by nitrogen flotation using 0.02 g/L of the enzyme Lafase XL Flot (Laffort, France) and 0.08 g/L vegetal protein Fitoproteina Xp (Vason, Trento, Italy). After flotation, the clear must with 50 to 150 NTU was moved to the fermentation tanks. The fermentation took place in three 125 L stainless steel deposits at 14 to 16 °C after inoculation with 0.015 g/L of *Saccharomyces cerevisiae* yeast (Vitilevure Chardonnay Yseo, Martin Vialate, Magenta, France). Fermentation was completed after twelve days (glucose plus fructose content lower than 0.5 g/L), and thereafter the wines were cold-settled, moved to 100 L stainless steel tanks, and sulfited (0.02 g/L). After two months, the wines were bottled and analyzed. The wines were named as CW (wines made by conventional winemaking).

For conventional winemaking with pre-fermentative maceration (PM), the destemmed and crushed grapes were taken to a 500 L stainless steel tank where 0.01 g/L of the pectolytic enzyme Lafase XL Press (Laffort, France) was added. Thereafter, the free-run must was moved to a 500 L tank and the skin mass was pressed in the pneumatic press (BucherVaslin XPro 8, France). Both free and press musts were mixed in the tank and quickly clarified by nitrogen flotation as previously described. The fermentation took place in three stainless steel deposits of 125 L at 14 to 16 °C after inoculation with 0.015 g/L of *S. cerevisiae* yeast (Vitilevure Chardonnay Yseo, Martin Vialate, Magenta, France). Fermentation was completed after twelve days (glucose plus fructose content lower than 0.5 g/L), and thereafter the wines were cold-settled, moved to 100 L stainless steel, and sulfited (0.02 g/L). After two months, the wines were bottled and analyzed. The wines were named as PM (wines made by pre-fermentative maceration).

For barrel fermentation (BF), the destemmed and crushed grapes were pressed as described for conventional winemaking, and the free run musts were also clarified by flotation as described for CW and PM. After flotation, the clear musts were moved to three new 225-L barrels of American oak and medium toast. The same yeast used in CW and PM was inoculated to carry out the alcoholic fermentation, which lasted thirteen days. After alcoholic fermentation, the wines were kept in the barrels for two months with periodic *battonages*. Thereafter, the wines were bottled and analyzed. The wines were named as BF (barrel fermentation).

2.2. Standard Enological Parameters

Standard oenological parameters in the wines were measured according to the official methods established by the International Organization of Vine and Wine [11]: pH, titratable acidity (g/L tartaric acid), volatile acidity (g/L acetic acid), and alcohol content (% vol: mL ethanol/100 mL wine). Malic acid and glucose plus fructose were analyzed by the autoanalyzer BioSystems Y15 (Biosystem, Barcelona, Spain).

2.3. Analysis of Volatile Compounds

The volatile composition was analyzed by gas chromatography-mass detector (GC-MS) after extraction by liquid–liquid according to [12,13]. Briefly, 8 mL of wine, 400 μL of dichloromethane, and 2.4 μg of internal standard (4-nonanol) were added to a flask. The extraction was carried out for 15 min with continuous stirring. Thereafter, the sample was centrifuged and the organic phase recovered. The aromatic extract was analyzed by GC-MS. Extraction of volatiles were made in triplicate.

A gas chromatograph (7890B) with an ion-trap mass spectrometer (7000C) by Agilent Technologies (Waldbronn, Germany) was used to analyze the volatile compounds. Injection was made into a capillary column CP-Wax 52 CB (50 m × 0.25 mm i.d., 0.2 μm film thickness, Chrompack (Agilent, Waldbronn, Germany). The temperature of the injector was programmed from 20 to 250 °C at 180 °C/min. The oven temperature was 40 °C, for 5 min, then programmed to rise from 40 to 250 °C, at 3 °C/min, then held 20 min at 250 °C and finally programmed to go from 250 to 255 °C at 1 °C/min. The carrier gas was helium N60 at 103 kPa, which corresponds to a linear speed of 180 cm/s at 150 °C and the split vent was set to 13 mL/min. The detector was set to electronic impact mode (70 eV), with an acquisition range from 29 to 360 m/z, and an acquisition rate of 610 ms.

Peaks identification was performed by comparison with mass spectra and retention times with those of pure standard compounds and confirmed by GC-MS. All of the compounds were quantified as 4-nonanol equivalents.

2.4. Odour Activity

To evaluate the contribution of a chemical compound to the aroma of a wine, the odor activity value (OAV) was determined. When OAV is higher than one, a possible contribution to the wine aroma is considered. OAV was calculated as the ratio between the concentration and the perception threshold of the individual compound. The perception threshold used in this work were those found in the literature [14–25].

2.5. Sensory Analysis

The sensory analysis was performed in a sensory room in accordance with ISO 8589 Standard (2010), and was carried out by thirteen expert tasters from the D.O.Ca Rioja (6 males and 7 females, 21–39 years old). In a first session, the tasters worked to stablish similar qualitative and quantitative criteria and to select a consensual group of descriptors. The specific training for this research consisted on tasting wines of the same variety and category (commercial samples). In a second session, wine samples were evaluated with a structured numerical scale of six points where 0 represented no intensity and 5 the highest intensity. The wines were presented in standard wine-tasting glasses in random order. Tasters rated the wines for visual, gustatory, and olfactory quality. The geometric mean (GM%) was calculated for each attribute as the square root of the product between relative intensity (I%) and relative frequency (F%). The wine descriptors were classified in basis to GM, according to the International Organization for Standardization ISO 11035 to make it possible to eliminate the descriptors with low GM.

2.6. Statistical Analysis

The statistical analyses were performed using XLstat-Premium (Addinsoft (2021). XLSTAT statistical and data analysis solution. New York, NY, USA. https://www.xlstat.com). One-way analysis of variance (ANOVA) with post-hoc Tukey HSD ($p < 0.05$) was made to determine the differences among treatments. The total degree of freedom of the experiments was two.

3. Results and Discussion

3.1. Oenological Parameters of Maturana Blanca Wines

Table 1 shows the ethanol content, pH, titratable acidity, volatile acidity, sum of glucose plus fructose, absorbance at 420 nm, and malic acid in Maturana Blanca wines elaborated with the different technologies.

The volatile acidity values indicated a suitable winemaking. All wines were fermented to dryness (glucose plus fructose content lower than 0.04 g/L). The values of ethanol degree were similar to those obtained in other monovarietal Spanish white wines [17,22,26,27]. However, the wines showed higher acidity and lower pH values than those described for these varieties, in good agreement with other studies [26], which describes Maturana Blanca wines as wines with low pH and high acidity. With the exception of the values of titratable acidity, the different elaborations did not produce significant differences in the standard oenological parameters. The titratable acidity was slightly lower in the wines with pre-fermentative maceration. Regarding the color, the Maturana wines showed higher values of yellowish tonalities (A = 420 nm) than those obtained in other monovarietal Spanish wines [27], and in good agreement with literature, which describes Maturana Blanca wines as greenish-yellow in color [10].

Table 1. Standard oenological parameters of Maturana Blanca wines produced by conventional winemaking (CW), conventional winemaking with pre-fermentative maceration (PM), and barrel fermentation (BF).

Parameters [1]	CW	PM	BF	F-Value
% Ethanol	13.16 ± 0.12	13.11 ± 0.10	13.19 ± 0.11	0.403 (ns)
TA	7.20 ± 0.10 b	6.70 ± 0.20 a	7.50 ± 0.20 b	16.333 **
pH	3.01 ± 0.05	3.03 ± 0.04	3.04 ± 0.02	0.467 (ns)
VA	0.20 ± 0.01 b	0.16 ± 0.01 a	0.31 ± 0.01 b	181.000 ***
G + F	0.03 ± 0.00	0.03 ± 0.01	0.02 ± 0.06	0.079 (ns)
MA	1.30 ± 0.02	1.40 ± 0.10	1.28 ± 0.05	2.884 (ns)
A 420 nm	0.29 ± 0.60	0.35 ± 0.30	0.31 ± 0.20	0.017 (ns)

[1] Ethanol: mL ethanol for 100 mL of wine; TA: titratable acidity as g tartaric acid/L; VA: volatile acidity as g acetic acid/L; G + F: sum of glucose plus fructose as g/L; MA: malic acid as g malic acid/L; Absorbance 420 nm. Different letters in the same line indicates statistically significant differences ($p < 0.05$) according to the Tukey HSD test ($n = 3$). Level of significance: ** and *** indicates significance at $p < 0.01$, and $p < 0.001$ respectively; ns: no significative statistical differences.

3.2. Wine Volatile Composition of Maturana Blanca Wines

The results obtained for the volatile composition of the Maturana Blanca wines made with conventional winemaking (CW), conventional winemaking with pre-fermentative maceration (PM), and barrel fermentation (BF), are shown in Table 2 and Figure 1. A total of 33 volatile compounds were detected by gas chromatography, and 26 of them were detected in all the wines (Table 2). The data have been organized into six chemical families: C6 alcohols, represented by 1-hexanol and E-3-hexenol; higher alcohols, represented by 2-methylpropanol, 1-octanol, 3-methyl-1-butanol, 4-methyl-1-pentanol, 3-methyl-1-pentanol, 2,3 butanediol, furfural, furfuryl alcohol, 3-methyltiopropanol, 2-phenylethanol, and benzyl alcohol; ethyl esters, represented by ethyl hexanoate, ethyl lactate, ethyl octanoate, ethyl decanoate, ethyl butyrate, diethyl succinate, and diethyl malate; acetates, represented by isoamyl acetate and 2-phenylacetate; volatile acids, represented by isobutyric acid, propanoic acid, hexanoic acid, octanoic acid, and decanoic acid; lactones, represented by γ-butirolactone, whiskey lactone, and γ-decalactone; carbonyl compounds, represented by acetoine; and volatile phenols, represented by 4-vinylphenol and eugenol. A one-way ANOVA was applied to test the effect of the winemaking on the individual volatile compounds. Figure 1 shows the data obtained arranged by volatile families. Table 3 shows the odor threshold and descriptor of each individual value.

Higher alcohols were quantitatively the largest group of the volatile compounds in all Maturana wines (Figure 1). Most of these compounds are synthesized by decarboxylation and reduction of α- keto-acids produced as intermediates of amino acid synthesis and catabolism during alcoholic fermentation. Some alcohols are related to herbaceous notes with strong and pungent tastes and smells, whereas others are positive contributors to wine aroma, being characterized by floral and fruity aromas (Table 3). Moreover, the family of furans, which includes furfural and furfuryl alcohol, is produced from wood polysaccharides by means of the Maillard reaction during the toasting process [28,29]. These compounds contribute to smoked and toasted nut notes [30]. In this study, all Maturana Blanca wines were characterized by high contents of 3-methyl-1-butanol and 2-phenylethanol. Both compounds are positive contributors to wine aroma and are characterized by banana notes [14] and floral and rose-like aromas [16]. The compounds 2-methylpropanol, characterized by fusel notes [14], and 2,3 butanediol, characterized by fruity aromas [14], were also found in significant concentrations, although both were below their odor thresholds (Table 3). The rest of higher alcohols showed concentrations below 100 µg/L.

Regarding the different winemaking technologies, the Maturana Blanca wines made with pre-fermentative maceration showed significantly higher concentrations of 3-methyl-1-butanol, 2,3 butanediol, and 2-phenylethanol, indicating that maceration with skins increased the amounts of higher alcohols contributing to floral and fruity aromas, in good agreement with other researches [6,31].

Table 2. Concentration (μg/L) of volatile compounds of Maturana Blanca wines produced by conventional winemaking (CW), conventional winemaking with pre-fermentative maceration (PM), and barrel fermentation (BF).

Compounds	CW		PM		BF		F-Value
	Mean	SD	Mean	SD	Mean	SD	
C6 alcohols							
1-hexanol	459.29	69.80	431.55	34.30	504.59	48.57	1.455 (ns)
E-3-hexenol	42.95	7.20	56.44	8.25	41.27	8.40	3.268 (ns)
Higher alcohols							
2-methylpropanol	990.51	13.63	809.63	79.22	978.53	118.14	4.510 (ns)
1-octanol	47.11	1.60	56.73	10.11	58.43	7.33	2.113 (ns)
3-methyl-1-butanol	30,856.85 a	1723.36	32,589.73 b	1642.59	26,179.37 a	1167.04	14.077 **
4-methyl-1-pentanol	31.08 b	3.81	24.27 a	1.70	25.83 ab	1.48	5.842 *
3-methyl-1-pentanol	67.03 b	7.73	45.88 a	2.16	59.31 ab	6.07	10.174 *
2,3 butanediol	169.85 ab	0.27	209.13 b	16.06	150.04 a	22.34	10.754 *
Furfural	nd	nd	365.43 a	77.32	2161.51 b	107.27	688.968 ***
Furfuryl alcohol	nd	nd	nd	nd	885.61	11.36	18,218.515 ***
3-methyltiopropanol	52.86 ab	0.23	47.82 a	2.04	56.86 b	5.58	5.228 *
2-phenylethanol	28,190.65 a	907.21	32,725.55 b	166.99	25,810.52 a	2957.88	11.570 **
Benzyl alcohol	17.00 a	2.00	21.64 a	4.17	nd	nd	54.604 ***
Ethyl esters							
Ethyl hexanoate	1153.15 a	234.02	1913.04 b	107.56	1077.51 a	70.22	26.968 **
Ethyl lactate	208.12 a	9.73	443.28 b	48.58	239.74 a	17.63	53.004 ***
Ethyl octanoate	1446.31 a	69.65	2335.61 b	189.27	1365.44 a	77.33	55.901 ***
Ethyl decanoate	285.27 a	35.40	361.25 b	27.20	290.42 a	11.70	7.618 *
Ethyl butyrate	325.12 a	22.70	425.42 b	33.70	285.89 a	15.40	24.681 **
Diethyl succinate	302.07 a	59.24	1166.60 b	46.75	308.57 a	12.95	379.650 ***
Diethyl malate	107.44 a	24.19	284.84 b	1.10	110.85 a	8.27	141.462 ***
Acetates							
Isoamyl acetate	3716.43 ab	273.39	4373.29 b	136.78	3021.53 a	485.73	12.485 **
2-Phenylacetate	878.77 a	58.26	1130.88 b	85.07	733.10 a	25.57	32.300 **
Volatile acids							
Isobutyric acid	nd	nd	185.94 a	32.41	442.55 b	11.52	375.648 ***
Propanoic acid	211.36 b	18.05	145.67 a	1.80	125.71 a	1.25	54.686 ***
Hexanoic acid	1634.83 a	195.31	1635.80 a	70.91	2320.43 b	52.75	30.641 **
Octanoic acid	5934.08	10.39	5978.48	334.44	5938.19	687.10	0.009 (ns)
Decanoic acid	199.34 a	20.54	120.40 a	11.22	1046.43 b	79.17	348.058 ***
Lactones							
γ-Butirolactone	195.90 a	23.88	185.94 a	32.41	nd	nd	67.625 ***
Whiskey lactone	nd	nd	nd	nd	442.03	40.48	357.773 ***
γ-Decalactone	176.84 a	22.79	187.40 a	14.50	372.19 b	8.74	134.756 ***
Carbonyl compounds							
Acetoine	70.08	6.65	72.27	4.41	76.68	2.18	1.486 (ns)
Volatile phenols							
4-Vinylphenol	505.35	228.46	229.01	55.25	416.50	162.08	2.197 (ns)
Eugenol	nd	nd	nd	nd	75.22	9.53	186.997 ***

Different letters in the same line indicates statistically significant differences ($p < 0.05$) according to the Tukey HSD test ($n = 3$). Level of significance: *, ** and *** indicates significance at $p < 0.05$, $p < 0.01$, and $p < 0.001$ respectively; ns: no significative statistical differences; nd: non-detected.

Table 3. Odor activity values (OAV) of Maturana Blanca wines produced by conventional winemaking (CW), conventional winemaking with pre-fermentative maceration (PM), and barrel fermentation (BF).

Compound	Odor Descriptor	Odor Threshold (µg/L)	Ref.	OAV			F-Value
				CW	PM	BF	
C6 alcohols							
1-hexanol	Green, cut grass	8000	[16]	0.06	0.05	0.06	1.455 (ns)
E-3-hexenol	Green, floral	400	[21]	0.11	0.14	0.10	3.268 (ns)
Higher alcohols							
2-methylpropanol	Fusel	40,000	[16]	0.02	0.02	0.02	4.510 (ns)
1-octanol	Rose, citrus	10,000	[23]	0.00	0.00	0.00	2.113 (ns)
3-methyl-1-butanol	Alcohol, banana	7000	[14]	**4.41** b	**4.65** b	**3.73** a	14.077 **
4-methyl-1-pentanol	Almond, toasted	5000	[21]	0.01 b	0.00 a	0.01 ab	5.842 *
3-methyl-1-pentanol	Herbaceous, cocoa	50,000	[21]	0.00 b	0.00 a	0.00 ab	10.174 *
2,3 butanediol	Fruity	150,000	[18]	0.00 ab	0.00 b	0.00 a	10.754 *
Furfural	Cocoa, smoky, nut	760	[25]	0.00 a	0.47 b	2.81 c	664.970 ***
Furfuryl alcohol	Cocoa, smoky, nut	2000	[24]	0.00 a	0.00 a	0.43 b	18,218.512 ***
3-methyltiopropanol	Cooked vegetable	1000	[18]	0.05 ab	0.05 a	0.06 b	5.228 *
2-phenylethanol	Floral, roses, lilac	10,000	[21]	**2.82** a	**3.27** b	**2.58** a	11.570 **
Benzyl alcohol	Caramel, fruity	200,000	[18]	0.00 b	0.00 b	0.00 a	54.604 ***
Ethyl esters							
Ethyl hexanoate	Apple, fruity	14	[17]	**82.37** a	**136.65** b	**76.97** a	26.968 **
Ethyl lactate	Strawberry, raspberry	154,000	[21]	0.00 a	0.00 b	0.00 a	53.004 ***
Ethyl octanoate	Apple, fruity	5	[17]	**289.26** a	**467.12** b	**273.09** a	55.901 ***
Ethyl decanoate	Grape	200	[21]	**1.42** a	**1.80** b	**1.45** a	7.618 *
Ethyl butyrate	Papaya, apple	20	[17]	**16.25** a	**21.25** b	**14.3** a	24.681 **
Diethyl succinate	Light fruity, wine	6000	[21]	0.05 a	0.19 b	0.05 a	379.650 ***
Diethyl malate	Over-ripe, peach, cut grass	760,000	[18]	0.00 a	0.00 b	0.00 a	141.461 ***
Acetates							
Isoamyl acetate	Banana, apple	30	[16]	**123.88** ab	**145.78** b	**100.72** a	12.485 **
2-Phenylacetate	Banana	250	[16]	**3.52** a	**4.52** b	**2.93** a	32.300 **
Volatile acids							
Isobutyric acid	Butter, cheese, rancid	2300	[15]	0.00 a	0.08 b	0.19 c	375.648 ***
Propanoic acid	Butter, rancid	8100	[18]	0.02 b	0.02 a	0.01 a	54.686 ***
Hexanoic acid	Cheese, fatty	3000	[21]	0.54 a	0.55 a	0.77 b	30.641 **
Octanoic acid	Cheese, fatty, rancid	1000	[21]	**5.93**	**5.98**	**5.94**	0.009 (ns)
Decanoic acid	Fatty, unpleasant, fat	10,000	[21]	0.02 a	0.01 a	0.10 b	348.058 ***
Lactones							
γ-Butirolactone	Toasty, wood, caramel	35,000	[22]	0.01 b	0.00 b	0.00 a	67.625 ***
Wisky lactone	Coconut, toast, wood	67	[14]	0.00 a	0.00 a	**6.60** b	357.773 ***
γ-Decalactone	Spicy	88	[14]	**2.01** a	**2.13** a	**4.23** b	134.756 ***
Carbonyl compounds							
Acetoine	Lactic	10,000	[19]	0.01	0.01	0.01	1.486 (ns)
Volatile phenols							
4-Vinylphenol	Smoky, almond	180	[15]	**2.25**	**1.27**	**2.73**	2.197 (ns)
Eugenol	Spices, clove, honey	6	[15]	0.00 a	0.00 a	**12.54** b	186.997 ***

Different letters in the same line indicates statistically significant differences ($p < 0.05$) according to the Tukey HSD test ($n = 3$). Level of significance: *, ** and *** indicates significance at $p < 0.05$, $p < 0.01$, and $p < 0.001$ respectively; ns: no significative statistical differences. The bold numbers indicate OAV values higher than one.

On the other hand, the wines made with fermentation in oak barrels showed the highest values of furans. Furfuryl alcohol was only detected in wines fermented in barrels, which also showed a high amount of furfural, both characterized by cocoa, nut, and smoky aromas [30]. Furfural is produced when pentoses (xylose) are heated and furfuryl alcohol is formed by enzymatic reduction of its analogous aldehyde during aging in oak barrels. The quantity of furfural in the wine depends, as well as on the age of the barrel, on the degree of wood toasting [28]. In the case of furfuryl alcohol, the important factors are those which affect the enzymatic activity such as the temperature and the pH [28]. Globally, in this study, the furfural concentration was much higher than that of furfuryl alcohol as it has been described that this compound extracts rapidly and reaches a maximum level after two months [32]. The concentration of furfuryl alcohol was in all cases lower than the odor threshold (Table 3).

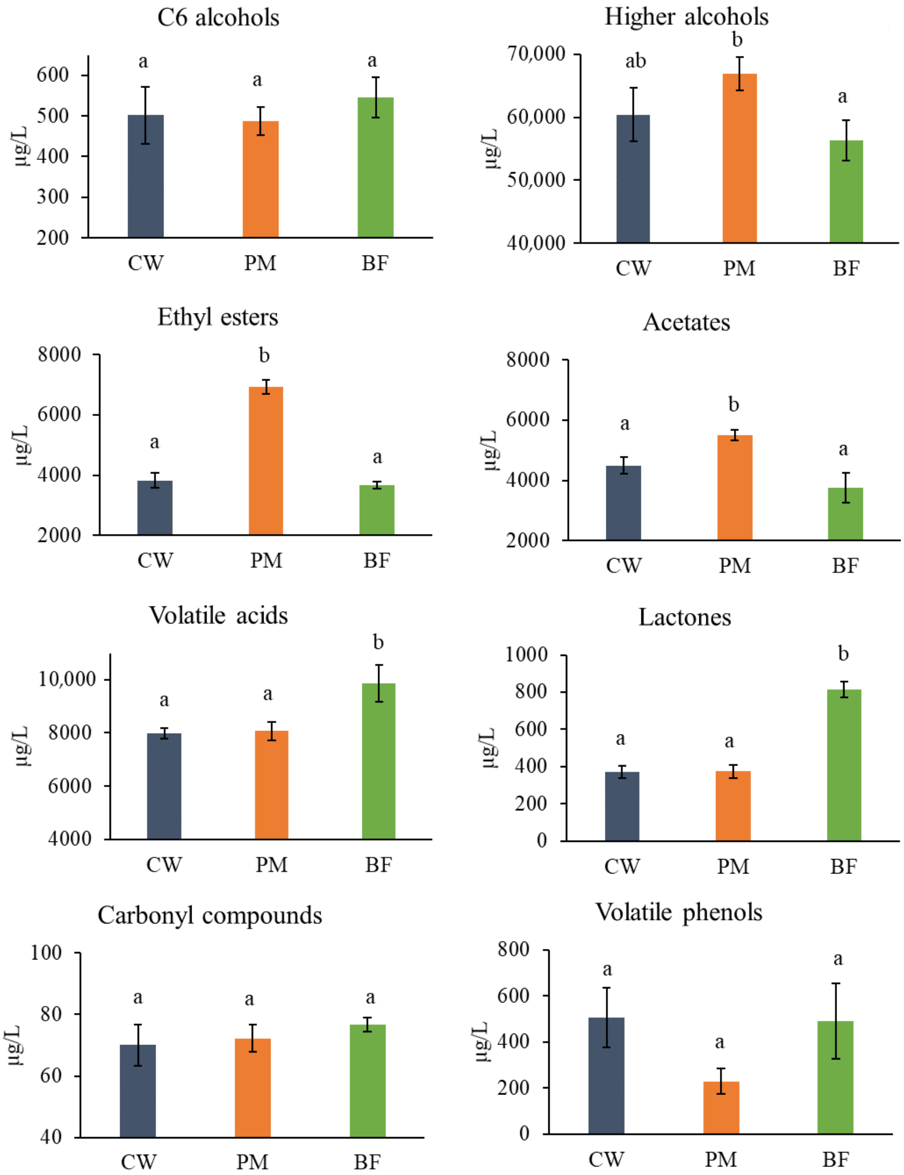

Figure 1. Concentration (µg/L) of volatile families of Maturana Blanca wines produced by conventional winemaking (CW), conventional winemaking with pre-fermentative maceration (PM) and barrel fermentation (BF). Different letters indicate statistically significant differences ($p < 0.05$) according to the Tukey HSD test ($n = 3$).

Acids were the second volatile family in quantity (Figure 1). This group of volatile compounds is produced by yeast and bacteria during fatty acid metabolism [33]. They show pungent, fatty, rancid, or cheesy odors (Table 3). Volatile fatty acids can contribute to the complexity of the wine bouquet even if present at sub-sensory threshold levels, while they have negative effect on wine aroma when above their thresholds [33]. Five volatile organic acids were detected in this study, but only octanoic acid showed a value above its odor (Table 3). Neither pre-fermentative maceration nor barrel fermentation affected the concentration of this compound.

Ethyl esters and acetates were found in important amounts in all the wines (Figure 1). Ethyl esters have a strong influence on wine aroma because they are usually found in high concentrations and have low detection thresholds (Table 3). They are especially important contributors to wine aroma because they are the primary source of fruity aromas. Most esters found in alcoholic beverages are secondary metabolites produced by yeast during alcoholic fermentation. Ethyl esters content depends on different factors, such as sugar content, fermentation temperature, aeration, and yeast strain [34]. In the present study, all the wines showed the same profile of ethyl esters of fatty acids. Ethyl hexanoate and ethyl octanoate were the major ethyl esters of fatty acids, followed by diethyl succinate, ethyl butyrate, ethyl lactate, ethyl decanoate, and finally diethyl malate. Ethyl hexanoate, ethyl octanoate, ethyl decanoate, and ethyl butyrate were detected above its odor threshold value in all the wines (Table 3). Pre-fermentative maceration significantly increased the content of total ethyl esters in the Maturana Blanca wines whereas barrel fermentation had no effect (Figure 1). All the ethyl esters significantly increased with pre-fermentative maceration, being diethyl succinate the compound most affected, with an increase of almost 400%, followed by diethyl malate, with an increase of 260%. Ethyl hexanoate, ethyl lactate, and ethyl octanoate almost doubled its concentration whereas ethyl octanoate increased 125%, in good agreement with other research, which also observed high increases in the concentrations of ethyl octanoate, ethyl decanoate, and diethyl succinate [6].

Acetates were also found in important quantities in all the wines (Figure 1). Acetate esters are produced from the reaction of acetyl-CoA with higher alcohols formed by degradation of amino acids or carbohydrates [33]. Two acetate esters were detected in this study (Table 2). Both isoamyl acetate [16], characterized by banana and apple aromas [35], and 2-phenylacetate, also characterized by banana notes, were detected in much higher quantities than their odor thresholds (Table 3), indicating that both were key molecules for Maturana Blanca aroma. As also observed in other studies [6,31,36], both compounds significantly increased with pre-fermentative maceration, 20% for isoamyl acetate and 30% for 2-phenylacetate. Fermentation in oak barrel aging did not affect the concentration of these compounds (Table 2 and Figure 1).

Only two C6 alcohols were identified in this study, 1-hexanol and E-3-hexenol (Table 2), with none above threshold (Table 3). The C6 alcohols usually add herbaceous and vegetal notes to grapes and wines, causing negative effect on wine aroma [27]. In this study, the amounts of these compounds were not affected by neither pre-fermentative maceration nor fermentation in oak barrels.

The family of volatile phenols were also detected in significant amounts in the wines (Figure 1). Two volatile phenols were identified in this study, 4-vinylphenol and eugenol (Table 2). In addition, 4-vinylphenol was found in the wines in similar quantities than that described for other white wines [27], as vinylphenols are the main phenols in white wines [27]. Eugenol was only detected in in barrel-fermented wines (Table 2). The family of volatile phenols coming from wood includes methylguaiacol, guaiacol, ethylphenol, vinylguaiacol, eugenol, ethylguaiacol, and trans-isoeugenol. Eugenol contributes to an interestingly spicy note of clove, while all the other volatile phenols contribute to smoked/toasted notes [37]. These volatile phenols originate from lignin during barrel toasting. Guaiacol and their derivatives form at high toasting temperatures [29] and since the barrels utilized in this study were submitted to medium toasting, they could not be detected. On the contrary, eugenol is found in untoasted oak [29] and its maximum extraction is reached in wines during the first months of contact with wood [38]. Therefore, the content of eugenol in wines fermented in oak barrels was above its threshold (Table 3).

Among the groups of lactones, three compounds were detected, γ-butirolactone, γ-decalactone, and whiskey lactone. γ-Butirolactone was detected in the wines in concentrations much lower than its odor threshold (Table 3). On the contrary, the concentration of γ-decalactone was very high in the wines fermented in barrels. Moreover, whiskey lactone was only detected in these wines and in concentrations much higher than its sensory threshold (Table 3). Among wine volatile compounds, lactones, and particularly the γ-lactones and whiskey lactones, play an important role in wine aroma. They are found in natural oak and are among the most important compounds contributing to the sensory properties of wines aged in wood. γ-Decalactone is described as spicy [19] or peach-like [20] while whiskey lactones are responsible for the coconut and toast flavor.

Finally, only acetoin was detected among carbonyl compounds, and, as expected in white wines, it was present in very low concentrations. The content of this compound was not affected by the winemaking technology applied.

3.3. Odour Activity Values of Maturana Blanca Wines

The importance of the volatile compounds depends on their concentration and on their olfactory perception thresholds, defined as the lowest concentration that can be detected by smelling. Therefore, the concentration/threshold ratio, known as odor activity value (OAV), shows the contribution of a specific compound to the wine aroma. Compounds with OAV values higher than 1 contribute to the wine aroma; however, the compounds with an OAV less than 1, might contribute to the aroma because of depressive or synergic effects with other compounds.

Table 3 shows the OAV of all the aromatic compounds analyzed. Data about their odor description and their perception threshold value are also shown in the same table. Fourteen of the volatile compounds identified in the Maturana Blanca wines presented OAV higher than 1 and, therefore, made a direct contribution to the aroma.

The compounds that directly contributed to the aroma of Maturana Blanca wines were those related to fruity (3-methyl-1-butanol, ethyl hexanoate, ethyl octanoate, ethyl decanoate, ethyl butyrate, isoamyl acetate, 2-phenylacetate), floral (2-phenylethanol), and spicy and smoke aromas (γ-decalactone, 4-vinylphenol), being all of them positive for the aroma of the wines. The majority volatile acids and hexanol showed concentrations lower than their perception threshold, which limits possible herbaceous and rancid and unpleasant nuances in the wines. The compounds that most contributed to the aroma of Maturana Blanca wines were the ethyl esters and acetates, and concretely those characterized by fruity aromas of banana and apple.

Comparing the different winemaking technologies, eleven compounds showed a direct contribution to the aroma in Maturana wines elaborated by conventional winemaking (CW) and conventional winemaking with pre-fermentative maceration (PM), while fourteen compounds showed OAV values higher than one in the wines fermented in oak barrels (BF). Regarding the total OAV, PM wines showed the highest value (OAV = 795.62), followed by BF wines (OAV = 512.9) and CW wines (OAV = 405.31). These results indicated that, a priori, Maturana wines fermented in barrels would originate more aromatic wines with more nuances than CW, and Maturana wines made with pre-fermentative maceration would produce the most aromatic wines.

The OAV of ethyl esters and acetates, with fruity nuances of apple and banana, significantly increased with pre-fermentative maceration, indicating higher contribution of these compounds to the wine flavor and aroma. Furfural, whisky lactone, and eugenol showed high OAV only in Maturana wines fermented in oak barrels, while they showed OAV less than one in the rest of the wines. These compounds come from wood and will contribute to aromas of cocoa, coconut, nut, toast, smoky, spices, and clove, increasing the aromatic complexity of these wines.

3.4. Sensory Properties of Maturana Blanca Wines

The sensory characterization of the Maturana Blanca wines was obtained from the attributes defined by the tasters. The geometric mean (GM%) was calculated for each attribute according to the ISO 11035 to make possible to eliminate the descriptors with low GM. The olfactory attributes of chemical, animal, and herbaceous showed GM values lower than <15% and were thus eliminated. The rest of the sensory attributes were used to define the sensory properties of Maturana Blanca wines (Figure 2). The intensity value shown corresponds to the mean value generated by all the tasters.

In the visual phase, Maturana Blanca wines were characterized by high values of color intensity, brightness, and yellowish tones, in good agreement with the data obtained for the oenological parameters and the descriptions found in other studies [10]. Except for the yellow tones, which were higher in the BF wines, no differences were observed among the different wines.

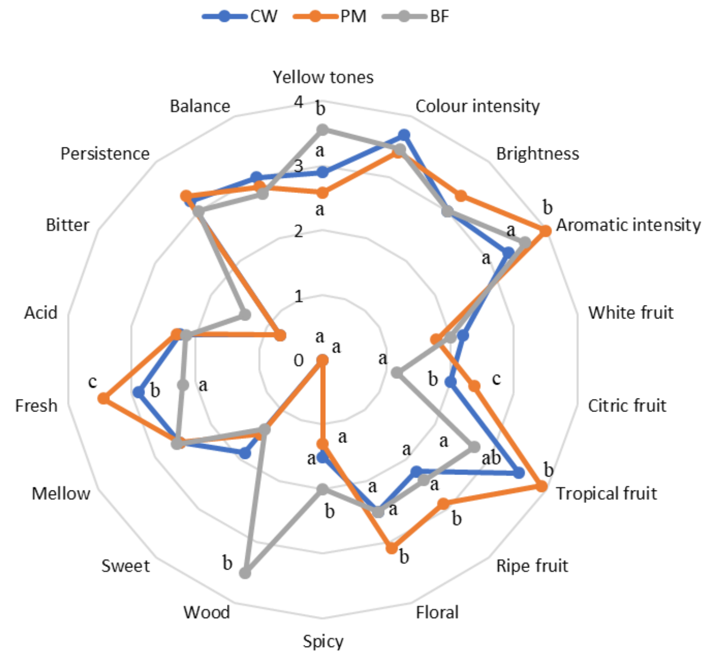

Figure 2. Sensory profile: visual, gustatory, and olfactory of Maturana Blanca wines produced by conventional winemaking (CW), conventional winemaking with pre-fermentative maceration (PM), and barrel fermentation (BF). Different letters indicate statistically significant differences ($p < 0.05$) according to the Tukey HSD test ($n = 3$).

In the olfactory phase, Maturana Blanca wines showed high aromatic intensity, with greater intensity of tropical and ripe fruit, and high values of floral aromas. Therefore, the descriptors with the highest GM were aromatic intensity, tropical fruit, ripe fruit, and floral, being 83%, 81%, 72%, and 77%, respectively. White and citric fruits, and spicy nuances were also detected by the tasters. On the contrary, herbaceous aromas could not be appreciated. Wines made with pre-fermentative maceration showed the highest aromatic intensity and greater values of floral aromas, ripe fruit, and tropical fruit, in good agreement with the data obtained in the instrumental analysis, where PM wines showed the highest value of total OAV, and significantly higher concentrations and OAV values of higher alcohols contributing to floral and fruity aromas, and ethyl esters and acetates, with fruity notes of apple and banana. Wines fermented in oak barrels showed the highest punctuations in the wood nuances and spicy notes. Hence, the sensory analysis again confirmed the results obtained in the analysis of volatile compounds as these wines showed high OAV values of furfural, whiskey lactone, and eugenol.

Finally, in the gustatory phase, Maturana Blanca wines were described as fresh and balanced in mouth, with notes of acidity and medium to high persistence.

4. Conclusions

Maturana Blanca wines were characterized by low pH values, high titratable acidity, and yellowish tonalities in color. A total of 33 volatile compounds were quantified by gas chromatography, which include C6 alcohols, higher alcohols, ethyl esters, acetates, volatile acids, lactones, carbonyl compounds, and volatile phenols. Fourteen compounds presented OAV values higher than 1 and, therefore, made a direct contribution to the aroma of Maturana Blanca wines. The compounds that most contributed to the aroma were those related to fruity (ethyl esters and acetates), floral aromas (2-phenylethanol), and spicy notes (γ-decalactone). Maturana Blanca wines were mainly characterized by volatile compounds of fruity aromas of banana and apple, in good agreement with the sensory analysis, were described as wines high aromatic intensity and predominance of tropical, ripe fruit, and floral aromas. Maturana Blanca wines were also characterized by high values of color intensity and yellowish tones, and were fresh and balanced in mouth, with slightly acidity and medium to high persistence.

Maturana Blanca wines made with pre-fermentative maceration showed the highest concentrations of ethyl esters and acetates, and produced wines with higher odor activity values, resulting in wines with greater aromatic intensity and greater values of floral aromas, ripe fruit, and tropical fruit. The aromatic profile of Maturana Blanca wines fermented in oak barrels showed higher complexity and they were also characterized by the presence of important amounts of furfural, whiskey lactone, and eugenol, resulting in wines with substantially higher wood and spicy nuances.

Author Contributions: Experimental design: B.A., Z.G., E.A.; methodology: B.A., Z.G., E.A.; winemaking: C.C., I.P., A.N.; software: L.M.-L.; statistical analysis: L.M.-L.; formal analysis: C.C., I.P., A.N.; writing—original draft preparation: Z.G.; writing—review and editing: B.A., Z.G.; supervision: Z.G., E.A.; project administration: B.A., Z.G., E.A. All authors have read and agreed to the published version of the manuscript.

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