



Article Quality, Nutritional, Volatile and Sensory Profiles and Consumer Acceptance of *Fondillón*, a Sustainable European Protected Wine

Hanán Issa-Issa ¹^(D), Francisca Hernández ²^(D), Leontina Lipan ¹^(D), David López-Lluch ³^(D) and Ángel A. Carbonell-Barrachina ^{1,*}^(D)

- ¹ Research Group "Food Quality and Safety", Centro de Investigación e Innovación Agroalimentaria y Agroambiental (CIAGRO-UMH), Miguel Hernández University, Carretera de Beniel, km 3.2, 03312 Orihuela, Spain; hissa@umh.es (H.I.-I.); leontina.lipan@goumh.umh.es (L.L.)
- ² Grupo de Investigación en Fruticultura y Técnicas de Producción, Centro de Investigación e Innovación Agroalimentaria y Agroambiental (CIAGRO-UMH), Miguel Hernández University, Carretera de Beniel, km 3.2, 03312 Orihuela, Spain; francisca.hernandez@umh.es
- ³ Departamento de Economía Agroambiental, Ingeniería Cartográfica y Expresión Gráfica en la Ingeniería, Centro de Investigación e Innovación Agroalimentaria y Agroambiental (CIAGRO-UMH), Miguel Hernández University, Carretera de Beniel, km 3.2, 03312 Orihuela, Spain; david.lopez@umh.es
- Correspondence: angel.carbonell@umh.es; Tel.: +34-6-0530-2372

Abstract: Sustainable irrigation strategies in Southeast Spain (one of the most arid regions in Europe) are essential to fight against desertification and climate change mitigation. In this way, *Fondillón* production is based on rain-based vineyards, over-ripe *Monastrell* grapes, and non-alcohol fortification. Thus, *Fondillón* is a naturally sweet red wine, protected within the Alicante Denomination of Origin, recognized by the European Union in its E-bachus database. The study aim was to evaluate the effect of the aging (*solera* factor) on *Fondillón:* (i) basic enological parameters (e.g., total, and volatile acidity), (ii) chromatic characteristics, (iii) antioxidant activity (ABTS^{•+}, FRAP and DPPH[•]), (iv) total contents of condensed tannins and anthocyanins, (v) volatile composition, (vi) sensory profile, and (vii) overall liking. Experimental data proved that the wine (1960 *solera*) with the highest total contents of condensed tannins and anthocyanins and total antioxidant activity was the most liked by Spanish consumers. Experimental results clearly established a positive relationship among *Fondillón* chemical composition, its antioxidant activity, and overall consumer liking. Exceptional harvest with grapes having extremely high antioxidant power (e.g., 1960 *solera*) will result, even more than 50 years later, in high quality wines with high consumer acceptance and a high monetary worth.

Keywords: affective sensory analysis; antioxidant activity; descriptive sensory analysis; DPPH•; phenolic content; rain-based farming; wine color

1. Introduction

Sustainability regarding environmental issues related to agriculture must be considered [1]. The coastal strip of the Southeast of Spain is one of the most arid regions of Europe, with an average rainfall of less than 400 mm [2]. There is still a need to promote public awareness, to truly reduce water consumption in the most demanding sectors, reduce uptakes form water bodies and, thus, the pressure made over them [3]. Thus, agriculture with low water requirement should be encouraged in arid and semi-arid regions rather than irrigation agriculture. In this sense, *Fondillón* vineyards located in Alicante (Valencian Community) are rain-fed and are fully hydroSOStainable, as defined by Sánchez-Bravo et al. [4]. The effect on preventing desertification through this crop is clear because vines are one of the few agricultural options due to the edaphic and climatic conditions in many areas. Moreover, encouraging the cultivation and production of this grape cultivar in the mentioned conditions, could be a way of increasing *Fondillón* production and profitability



Citation: Issa-Issa, H.; Hernández, F.; Lipan, L.; López-Lluch, D.; Carbonell-Barrachina, Á.A. Quality, Nutritional, Volatile and Sensory Profiles and Consumer Acceptance of *Fondillón*, a Sustainable European Protected Wine. *Agronomy* **2021**, *11*, 1701. https://doi.org/10.3390/ agronomy11091701

Academic Editor: Dugald C. Close

Received: 9 August 2021 Accepted: 22 August 2021 Published: 26 August 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). as well as creating jobs and value in rural areas. As explained by other authors, this can be a successful tool to mitigate climate change in arid and semiarid regions considering adequate farm and cultivation practices and systems [5].

Fondillón is a strong *rancio* wine from Alicante made from *Monastrell* grape and matured like an Oloroso sherry (oxidized for a long aging period in oak barrels; minimum of 10 years). The main difference with Oloroso sherry is that *Fondillón* is not fortified. Thus, *Fondillón* is a naturally sweet wine (included by the European Union in its E-Bacchus database) produced in the Alicante Protected Designation of Origin, Alicante PDO [6]. Naturally sweet wines come from overripe and or dehydrated grapes, are non-fortified wines, and have a total alcoholic strength of not less than 15% by volume (abv) [7].

Rancio is an imprecise tasting term used in many languages for a distinctive style of wine achieved by deliberately maderizing the wine by exposing it (i) to oxygen and/or (ii) heat. Key flavor compounds identified in aged *vin doux naturels* arise by Maillard reaction of sugars with amino acids and by oxidation [8]. Maderization implies mild oxidation over a long period of time and sometimes heat. It should be properly applied only to wines with (i) a high enough alcoholic strength to inhibit the action of *Agrobacter*, which would otherwise transform the wine into vinegar, and (ii) a high antioxidant capacity, which is needed to preserve the maximum and guarantee that the transformation of aroma precursors into the proper volatile compounds is achieved without generation of any type of off-flavors.

Very few maderized wines are made today by simply ageing the wine at cellar temperature; the oxidation process is instead hastened by heating or "baking" the wine as on the Madeira Island. Oxidation reactions, like most organic chemical reactions, can be roughly doubled in speed by a temperature rise of 10 °C [9,10]. A high level of oxidation is a key part of the style of fortified wines (such as Oloroso Sherry or Madeira); the oxidation is evident from the brown color of the wine and a lack of fresh fruit on the palate, which is instead replaced by nuts and dried fruits. In this case, the wine is stored for years in barrels that are not filled to the top, allowing oxygen within the headspace of the barrel (ullage). More commonly, for un-fortified red wines, the exposure to oxygen is limited and therefore has a much slower, subtler effect.

Aging and oxidation will affect wine composition [11]. However, changes with time of chromatic characteristics, antioxidant activity, and total polyphenolic composition under oxidation conditions in oak barrels for a long period of time without fortifying and/or heating the wine are not well known. Consequently, the aim of this study was to evaluate in *Fondillón* wine the effect of the aging (*solera* factor) on (i) basic enological parameters (e.g., total and volatile acidity), (ii) chromatic characteristics, (iii) antioxidant activity (ABTS^{•+}, FRAP, and DPPH[•]), (iv) total contents of condensed tannins and anthocyanins, (v) volatile composition, (vi) sensory profile, and (vii) overall liking of *Fondillón* consumers.

2. Materials and Methods

2.1. Wine Samples and Experimental Design

The factor under study was "*solera*" (aging time) and included nine levels: 1930, 1944, 1950, 1960, 1969, 1975, 1980, 1987, and 1996. The samples used (nine bottles of 500 mL per *solera*) were kindly supplied by Bodegas Monóvar-MGWines (Monóvar, Alicante, Spain). The samples were transported and stored under optimal conditions at the facilities of the Miguel Hernández University (Orihuela, Alicante, Spain) to later carry out the corresponding analyses. All analyses were run, at least, in triplicate, using different bottles for each replication; this is, samples from three bottles were taken and analyzed.

2.2. Enological Parameters

The basic enological parameters [pH, relative density, total alcohol content (% vol.), total acidity (g tartaric acid L^{-1}), and volatile acidity (g acetic acid L^{-1})] were determined, in triplicate, according to the International Organization of Vine and Wine Compendium methods of analysis [12].

2.3. Chromatic Characteristics, Total Polyphenol Index (TPI), Total Anthocyanin Content (TA), Antioxidant Activity (AA) and Total Condensed Tannins (TCT)

<u>The chromatic characteristics</u>: (i) color intensity, (ii) tonality, and (iii) color density in the wine samples were determined according to methods OIV-MA-AS2-07B [13] and Glories [13] using a spectrophotometer (ThermoSpectronic He γ ios γ , Cambridge, England). All analyses were carried out in triplicate.

The color intensity (CI) was calculated as the sum of the absorbance at 420, 520, and 620 nm (CI = $A_{420} + A_{520} + A_{620}$). The tonality (T) was calculated as the ratio of absorbance at 420 nm to absorbance at 520 nm (T = A_{420}/A_{520}).

The color density (CD) was calculated as the sum of the absorbance at 420 and 520 nm (CD = $A_{420} + A_{520}$). Also, Glories colorimetric index [14] percentages of the colors yellow, red, and blue were obtained using the absorbance values at 420, 520, and 620 nm as follows:

- Yellow, Y (%) = $(A_{420}/CI) \times 100$
- Red, R (%) = $(A_{520}/CI) \times 100$
- Blue, B (%) = $(A_{620}/CI) \times 100$

Total polyphenol index (TPI) was evaluated by measuring the absorbance at 280 nm on a UV–visible spectrophotometer (Helios Gamma model, UVG 1002E) [15]. The TPI was determined after diluting the wine sample 100 times. Analyses were run in triplicate.

<u>Total anthocyanin content (TA)</u> was determined according to Di Stefano et al. [16]. Briefly, the absorbance of the samples was measured at 540 nm using a UV–visible spectrophotometer (Helios Gamma model, UVG 1002E) and the total anthocyanin content was calculated using the formula by Di Stefano et al. [16]. Analysis was run in triplicate, and results were expressed as equivalents of malvidin 3-O-glucoside (mg L⁻¹).

Antioxidant activity (AA) was evaluated using three methods:

- (i) The free radical scavenging capacity using the ABTS^{+•} [2,2'-azino-bis (3-ethylbenzothi azoline-6-sulphonic acid)] method described by Re et al. [17], with absorbance being measured at 734 nm (UV-visible spectrophotometer, Helios Gamma model, UVG 1002E). Calibration curve (3.5–5.0 mmol Trolox L⁻¹) with good linearity ($R^2 \ge 0.999$) was used for the quantification. Analyses were run in triplicate and results were expressed as mmol Trolox L⁻¹.
- (ii) The free radical scavenging capacity was also measured using the DPPH[•] method as proposed by Katalinic et al. [18], with absorbance being measured at 517 nm. The inhibition percentage of the DPPH[•] radical was determined according to the following Equation (1):

$$\left[\frac{AC(0) - AA(t)}{AC(0)}\right] \times 100\tag{1}$$

where AC(0) is the absorbance of the sample at t = 0 min, and AA(t) is the absorbance of sample at t = 16 min. Analyses were carried out in triplicate.

(iii) The ferric reducing/antioxidant power (FRAP) was performed on a modified version of the method by Benzie et al. [19]. It is based on the reducing power of antioxidants, which will reduce the Fe^{3+} to Fe^{2+} in the form of a blue complex ($Fe^{2+}/TPTZ$). Absorbance was measured at 593 m. Analyses were run in triplicate and results were expressed as mmol Trolox L^{-1} .

<u>Total condensed tannins (TCT)</u> were determined according to Ribéreau-Gayon and Stonestreet [20]. Briefly, the samples were prepared in two test tubes, one for the control (tube 2) and the other for the hydrolysis (tube 1); in this tube, 4 mL of wine diluted in 1:50 distilled water, 2 mL of distilled water, and 6 mL of HCl 12 N were added successively. Subsequently, the closed hydrolysis tube was placed in a water bath at 100 °C for 30 min, and then cooled in an ice water bath. Then, 1 mL of 95% ethanol was added to the two tubes to solubilize the red color that appeared and finally the absorbance (Abs) of the two tubes was measured at 550 nm under an optical path of 1 cm, using water as a reference.

Tannins were determined by the following equation: Tannins (g L^{-1}) = 19.33 × (Abs tube 1–Abs tube 2). The coefficient of 19.33 corresponds to the molar extinction coefficient of the cyanidin obtained by the acid hydrolysis of the condensed tannins, corrected to directly give the result in g L^{-1} .

2.4. Volatile Compounds

The extraction of volatile compounds was done by HS-SPME technique with 1 cm DVB/CAR/PDMS fiber (Supelco, Bellegonte, PA, USA). Briefly, 10 mL of sample were placed into 20 mL headspace vial together with 5 μ L of benzyl acetate (1000 mg L⁻¹) as internal standard and 1 g of NaCl. Thereafter, the sample was placed on autosampler (AOC-6000 Plus, Shimadzu) at 250 rpm and 40 °C, for 40 min. The identification and quantification of the volatile compounds was carried out using a Shimadzu GC2030 gas chromatograph and a TQ8040 NX triple quadrupole mass spectrometer as detector (Shimadzu Scientific Instruments, Inc., Columbia, MD, USA) equipped with an AOC-6000 Plus autosampler. Only the single quadrupole acquisition mode was exploited on the TQ8040 NX (Q3 Scan). Data were acquired by using the GCMS solution software ver. 4.4 (Shimadzu). The column used was an X5MS (silphenylene polymer; Teknokroma, Barcelona, Spain) with dimensions of 30 m (length), 0.25 mm (internal diameter), and 0.25 μ m (film thickness). The GC temperature program was, as follows: 50 °C hold for 2 min, then +3 °C min⁻¹ ramp up to 170 °C, and an increment of 20 °C min⁻¹ up to 230 °C. Helium head pressure was 18 kPa (constant linear velocity mode 30 cm s⁻¹). Injector, ion source, and interface were at 230, 230, and 280 °C, respectively. Helium with flow 0.6 mL min⁻¹ was used as carrier gas with a split ratio of 1:10. Volatile compounds were identified by comparison of: (i) experimentally obtained mass spectra with those available in NIST 17 Mass Spectral, and (ii) linear retention indices calculated using the C6-C20 n-alkane mix (sigma-Aldrich, Steinheim, Germany). Only compounds with spectra similarity >90% were considered as correct hits; besides, the linear retention similarity threshold was set up at ± 10 units. Analyses were conducted in triplicate and results were expressed as mg L^{-1} .

2.5. Descriptive Sensory Analysis with Trained Panel

A trained panel, consisting of eight panelists (four women and four men), aged between 35 and 60 years, was used to conduct the descriptive sensory analysis of the *Fondillón* samples under study. This panel had more than 300 h of experience, it belongs to the Regulatory Council of the Wines of Alicante Protected Designation of Origin (DOP Alicante), was trained according to ISO 8586:2012 [21] and is certified by the National Accreditation Agency (ENAC) under the ISO 17065 [22].

The attributes and vocabulary used by the panelists to evaluate the *Fondillón* samples were those found in the official lexicon of the DOP Alicante [6]. The attributes under evaluation were: (i) appearance: color; (ii) odor (alcohol, fruity, floral, Mediterranean forest, spicy, animal, toasted, and chemical); (iii) basic tastes: sweetness, sourness, and bitterness; (iv) flavor: alcohol, fruity, floral, Mediterranean forest, spicy, animal, toasted, chemical, and aftertaste); and (v) chemical sensations (astringent). The panelists used a structure 10-point scale (0 was extremely low or no intensity, and 10 was extremely high intensity), with increments of 0.5 units, to evaluate the intensity of each of the attributes.

The nine samples under analysis were run in triplicate in four sessions of ~2.0 h. In each of the sessions, two quality control parameters were included to evaluate the panel performance: (i) reproducibility (same sample evaluated in two different sessions) and (ii) repeatability (same sample evaluated twice in the same session); to validate the panel performance, the deviation of these two parameters must be below 20% for all sensory descriptors. A maximum of 10 samples can be evaluated in a single session of 2.0 h, including three quality control samples (reproducibility, repeatability, and sample with a defect). Samples were randomly served coded with a three-digit numbers, together with the appropriate questionnaire, one at a time, and waiting 10 min between samples. Between samples and for palate cleansing, water and unsalted crackers were provided

to panelists. For the descriptive analysis, each panelist was initially provided with a black cup with 35 mL of the sample for the analysis of the odor, basis tastes, flavor and chemical sensations, and later to a transparent cup with 20 mL for the color analysis, at a temperature of 16–18 °C, breadsticks and water were also provided to clean the palate between samples. This evaluation was carried out in normalized sensory booths with white light at a temperature of 22 ± 2 °C.

2.6. Affective Sensory Analysis

The affective sensory analysis was carried out with 123 Spanish consumers, which were recruited from the Province of Alicante. The key recruitment requirement was that consumers should be regular *Fondillón* or oxidized wines consumers. The study was developed according to a balanced incomplete block design (split-plot), with participants testing five of the nine *Fondillón* samples. The samples were served to each consumer under the same tasting conditions (wine temperature, wine volume, and palate cleansers) previously mentioned for the descriptive study, in a random order, and coded with three-digit codes. Overall liking together with the satisfaction degree of the sample sweetness and aftertaste were evaluated, using a 9-point hedonic scale: 1 = dislike extremely; 5 = neither like or dislike; and 9 = like extremely. Finally, questions were asked about wine preference and purchase intention for the samples under study (Table S1).

2.7. Statistical Analysis

The results were first subjected to a one-way analysis of variance (ANOVA) using the *solera* age as the factor, and later to the Tukey or LSD (descriptive and affective sensory data) multiple range test. For the sensory data, a requirement of the ENAC-certified sensory panel is that the factor "judge" must be preliminary analyzed to ensure that it has no significant effect on the results of the panel. If any of the panelists produce significantly different results, its data must be removed, and the statistical analysis must be redone. Then, the factor or factors under study (e.g., *solera*) is considered. Differences were considered statistically significant at p < 0.05. To perform these statistical analyses, the software XLSTAT Premium 2016 (Addinsoft, New York, NY, USA) and Statgraphics Plus (version 3.1, Statistical Graphics Corp., Rockville, MA, USA) were used.

3. Results and Discussion

3.1. Enological Parameters

The legislation of the DOP Alicante establishes several thresholds for the physicochemical parameters in *Fondillón* samples. In this way, the total acidity must be above 3.50 g of tartaric acid L^{-1} , the volatile acidity must be below 1.50 g of acetic acid L^{-1} , and the total alcohol content must be above 16% v/v [23]. Regarding the present results, the factor "*solera*" did not significantly affect either the pH or the relative density; however, there were significant effects on the rest of parameters, although without a clear trend with the "*solera*" age (Table 1).

	лU	Relative Density (20 $^{\circ}$ C)	Total Alcohol Content	Total Acidity	Volatile Acidity							
	pn	(g mL ⁻¹)	(% <i>v</i> / <i>v</i>)	(g tartaric acid L^{-1})	(g acetic acid L^{-1})							
Solera		ANOVA ⁺										
	NS	NS	**	**	***							
			Tukey Multiple Range T	est ‡								
1930	3.55	0.9967	21.17 a	8.63 ab	1.16 cd							
1944	3.67	1.0098	20.46 ab	8.40 ab	1.46 ab							
1950	3.59	0.9972	19.30 ab	8.63 ab	1.13 d							
1960	3.44	0.9985	19.60 ab	10.01 a	1.31 bc							
1969	3.68	0.9978	18.53 ab	9.00 ab	1.37 ab							
1975	3.82	1.0000	18.93 ab	7.80 b	1.01 d							

Table 1. Basic enological parameters of *Fondillón* wines as affected by the "solera" factor.

	- U	Relative Density (20 $^{\circ}$ C)	Total Alcohol Content	Total Acidity	Volatile Acidity						
	рп	(g mL ⁻¹)	(% <i>v/v</i>)	(g tartaric acid L^{-1})	(g acetic acid L^{-1})						
Solera	ANOVA ⁺										
	NS	NS	**	**	***						
	Tukey Multiple Range Test ‡										
1980	3.35	0.9959	18.90 ab	8.33 ab	1.50 a						
1987	3.51	0.9984	18.44 ab	7.65 b	1.13 cd						
1996	3.43	0.9970	18.30 b	7.28 b	1.16 cd						
Minimum	3.35	0.9959	18.30	7.28	1.01						
Maximum	3.82	1.0098	21.17	10.01	1.50						
Legal threshold 4			>16	>3.5	<1.5						

Table 1. Cont.

⁺ NS = not significant at p < 0.05; ** and *** significant at p < 0.01, and 0.001, respectively. [‡] Values (mean of three replications) followed by the same letter, within the same column, were not significantly different (p < 0.05), according to Tukey's least significant difference test [¥] [20].

The pH plays an important role in the stability and sensory quality of the wine as it is linked to longevity, aroma, and color [24]. The pH of red wines must range between 3.30 and 3.60, because above these values the wines could have a flat flavor and on the contrary, below this threshold the wines would be harsh and could be unpleasant for consumption [21]. The pH of the *Fondillón* samples under study ranged between 3.35 (*solera* 1980) and 3.82 (*solera* 1975) and presented a mean value of 3.56 (Table 1). Thus, these values are within the previously mentioned parameters to meet the normality range, except for three values, which were slightly above the legal thresholds, perhaps implying a low negative effect of the aging process. Nogueira et al. [25] studied the effect of (i) type and (ii) age of on the main physico-chemical parameters of Madeira wines and concluded that their pH was unaffected by these two factors and ranged between 3.26 and 3.42.

Acidity is one of the most important characteristics to be controlled when making a wine. The total acidity is the result of the sum of all the organic acids present in the wine and has a direct and positive influence on the conservation of the wine, because it inhibits the development of microorganisms. Volatile acidity is another important parameter in wines, because above a threshold it shows deterioration of the wine due to production of acetic acid during yeast fermentation or also during aging in oak barrels by the chemical hydrolysis of hemicellulose [26]. The experimental ranges for these two parameters were: (i) 7.28 (*solera* 1996) and 10.01 g tartaric acid L^{-1} (*solera* 1960), and (ii) 1.01 (*solera* 1975) to 1.50 g acetic acid L^{-1} (*solera* 1980), respectively (Table 1).

Consequently, all experimental values of the total and volatile acidities were above and below the legal thresholds for *Fondillón* wines, respectively, and can be declared as appropriate; the sample *solera* 1980 was just in the legal threshold for the volatile acidity.

Nogueira et al. [25] also reported that total and volatile acidity seem to increase with aging for all types of Madeira wines, for samples up to 10 years old, and then, the contents were stable and below the maximum concentration admissible, for example 1.2 g L⁻¹ for the volatile acidity. It is important to consider that the *Fondillón* samples under analysis in the current study were around 25 years old, and the reported increase in these two parameters in the Madeira wines could have happened at the beginning of the aging period also in the *Fondillón*, but the values reported in the current study are representative of the final steady state. This is a possible explanation for the fact that the "*solera*" factor did not affect the values of these enological parameters.

Regarding the alcohol content, it is reported that it must have at least 16% and it is of the outmost importance to remark that "no fortification can be done" [23]. The alcoholic content of the analyzed wines ranged between 18.30 (*solera* 1996) and 21.17% v/v (*solera* 1930), with a mean of 19.28% (Table 1). A general trend was found, with alcohol content increasing with the wine *solera* (R² = 0.848; p < 0.01). A study carried out by Gómes-Cebrián [27], on the aromatic characterization of the *Fondillón* samples being marketed on 2013 (10 brands), showed that the mean alcohol content was 16.65% v/v, with values

ranging between 15.7 and 18.10%. The values found in the current study are higher than those reported in the older study, and this might occur due to the age of the studied wines. Nogueira et al. [25] found that the average alcoholic content for young Madeira wines was up to 18%, while a slight increase above 19% was reported for 10-year-old wines, although no statistical support was provided. It is important to remember that Madeira wines are "fortified", reaching alcoholic contents in the range 15–22% [28], while the alcoholic content of *Fondillón* is only due to the fermentation of the natural sugars of Monastrell grapes by the yeast.

As a conclusion for this section, it can be stated that all *Fondillón* samples under analysis were within the legal limits established by the regulation of the DOP Alicante, and the physico-chemical profile of *Fondillón* seems not to be affected by the "*solera*" factor due to the long aging time of the wines, above 25 years.

3.2. Chromatic Characteristics, Total Polyphenol Index (TPI), Total Anthocyanin Content (TA), Antioxidant Activity (AA) and Total Condensed Tannins (TCT)

Color is one of the most important organoleptic aspects of a wine, as it can be an indicator of quality and can influence consumer acceptance and its satisfaction degree. This attribute depends mainly on (i) the grape type and enological parameters at harvest, (ii) vinification processes, and (iii) aging type and time [29].

The hue and intensity of the color are parameters mainly linked to the content of anthocyanins, which are extracted from the grape during maceration. Anthocyanins have a high antioxidant capacity, which are very beneficial for people's health [30]. During *Fondillón* oxidative aging, anthocyanins undergo different oxidation reactions such as condensation and polymerization, producing changes in the initial bluish tones transforming them into orange [31].

The parameter "tonality" (Y/R) measures the relationship between the intensity of the yellow component, Y (420 nm), and red one, R (520 nm). In the studied *Fondillón* samples, significant positive and negative correlations were observed among the tonality and (i) the Y component ($R^2 = 0.987$; p < 0.001) and (ii) R component ($R^2 = 0.982$; p < 0.001), respectively (Figure 1a). These relationships meant that during aging there was an increase in the Y component and a decrease in the R component, leading to an increase in the wine tonality. These results agreed with those reported by Del Fresno et al. [32] in their study of the changes of phenolic fraction of red wines (cultivar Tempranillo) aged in oak barrels.

Regarding the parameter "color intensity", a positively correlation was observed with TPI ($\mathbb{R}^2 = 0.690$; p < 0.01) and the TA ($\mathbb{R}^2 = 0.757$; p < 0.01) (Figure 1b,c). These correlations meant that a high color intensity was linked to a high content of polyphenols and anthocyanins. In this way, the *Fondillón* sample with the highest values of color intensity (5.49), color density (4.80), R component (33.3), B component (12.5), and TA (0.76 mg L⁻¹) was that of the 1960 *solera*, which at the same time had the lowest values of the tonality (1.62) and Y component (54.1) (Table 2); all these values made *Fondillón solera* 1960 the most special one within the studied wine collection.

The presence of phenolic compounds in wines has a direct effect on their antioxidant activity. The most important phenolics in wine are flavonoids (anthocyanins, flavanols and flavanols), which play a key role in the wine antioxidant activity and sensory profile, by influencing color, astringency, and bitterness [33]. In this way, a significant positive correlation was found between TPI and TCT ($\mathbb{R}^2 = 0.615$; p < 0.05); however, no other significant correlations were found among TPI, TCT, and TA (Table S2).

For the analysis of the antioxidant activity, three methods were used: ABTS⁺⁺, DPPH⁺, and FRAP, because each one of them has different mechanisms of action. Table 2 shows that the *Fondillón* that presented the highest value for the three methods again was that of the *solera* 1960: ABTS⁺⁺ (2.02 mmol Trolox kg⁻¹), FRAP (4.97 mmol Trolox kg⁻¹), and DPPH[•] (64.4%), with the *solera* 1996 (the youngest *Fondillón* under study) being on the opposite side (lowest AA values): ABTS⁺⁺ (1.28 mmol Trolox kg⁻¹), FRAP (4.28 mmol Trolox kg⁻¹), and finally DPPH[•] (29.9%). It was also observed that there were significant positive correlations among the TA and ABTS^{•+} ($R^2 = 0.645$; p < 0.01) and DPPH[•] ($R^2 = 0.640$; p < 0.01) (Figure 1d); no significant effect was observed on FRAP. These results agreed with those presented by Luna et al. [15], when they studied the phenolic composition and antioxidant activity of minor grape varieties (Sabater, Gorgollassa, Escursac, Giró Ros, and Quigat) from the Balearic Islands (Spain) and concluded that the higher the total phenolic content, the higher the antioxidant activity.

The current experimental data showed a general trend of an increase in the DPPH• with the aging time ($R^2 = 0.224$; p < 0.05). No clear trend was found for the ABTS^{•+} or FRAP data. The DPPH[•] trend found in *Fondillón* agreed with the results published by Larrauri et al. [34], who reported higher values of DPPH[•] for older Spanish wines prepared using Tempranillo grapes from fours Appellation of Origin (Madrid, Rioja, Ribera de Duero, and Valdepeñas). Later, contradictory results regarding the antioxidant activity were reported by Rivero-Pérez et al. [35], who studied 162 samples of red wine from Castilla y León. Their FRAP data showed an increase with the aging time, perhaps due to the transfer of ellagitannins from the wood to the wine in the first stages of aging; however, the opposite trend was found for ABTS⁺⁺ and DPPH[•] data, with values decreasing with the aging time. This behavior could be justified because these two last methods have the same antioxidant mechanism, that is, single-electron transfer mechanism [35]. Another possible explication for the increase in antioxidant activity with the aging time found in different wine types, including some Fondillón soleras, could be the formation of polar products due to Maillard reactions happening during wine storage [36,37]. In this way, Moreno et al. [36] studied the antioxidant activity of Pedro Ximenez musts and reported an increase in the antioxidant values linked to Maillard reactions.



Figure 1. Correlation between color composition (*Y*, *R*) and tonality (**a**); correlation between color intensity and phenolics index (**b**); correlation between color intensity and total anthocyanins (**c**); and correlation between total anthocyanins content and antioxidant activity ($ABTS^{\bullet+}$, $DPPH^{\bullet}$) (**d**).

Solera I	Color	Tonality	Color	Υ¶	R ¶	$B^{ \P}$	ABTS•+	FRAP	DPPH•	Total Phenolic	Total Condensed Tannins Content, TCT	Total Anthocyanin Content, TA
	Intensity	5	Density		(%)		(mmol Trolox kg ⁻¹)		(%)	– Index, TPI	(g L ⁻¹)	(mg L ⁻¹)
								ANOVA ⁺				
	***	***	***	***	***	***	***	**	***	***	***	***
Tukey Multiple Range Test ‡												
1930	4.03 cd	2.30 c	3.72 bcd	64.2 b	27.9 d	7.88 f	1.63 c	4.61 ab	45.3 e	14.5 b	0.60 bc	0.53 ef
1944	3.69 d	2.46 ab	3.43 cd	66.2 a	26.9 e	6.96 g	1.82 b	4.48 ab	49.7 cd	14.2 bc	1.24 a	0.54 ef
1950	4.81 ab	2.01 de	4.27 ab	59.3 d	29.5 c	11.3 b	1.78 b	4.54 ab	51.6 bc	17.7 a	1.07 a	0.69 abc
1960	5.49 a	1.62 f	4.80 a	54.1 e	33.3 a	12.5 a	2.02 a	4.97 a	64.4 a	17.0 a	0.98 ab	0.76 a
1969	3.73 d	2.37 bc	3.42 d	64.5 b	27.2 de	8.29 e	1.49 d	4.37 ab	37.9 f	14.4 b	0.38 cd	0.62 cde
1975	3.79 cd	2.49 a	3.52 cd	66.2 a	26.6 e	7.14 g	1.73 bc	4.15 b	48.8 d	12.5 bcd	0.45 cd	0.65 bcd
1980	4.50 bc	1.97 e	4.07 bc	60.0 d	30.5 b	9.46 d	1.98 a	4.91 ab	52.3 b	13.8 bc	0.61 bc	0.73 ab
1987	4.10 bcd	2.12 d	3.70 bcd	61.2 c	28.9 с	9.93 c	1.63 c	4.47 ab	38.5 f	12.0 cd	0.18 d	0.59 de
1996	3.16 d	2.45 abc	2.90 d	65.2 ab	26.6 e	8.14 ef	1.28 e	4.28 ab	29.0 g	10.6 d	0.10 d	0.48 f

Table 2. Chromatic Characteristics, antioxidant activity, total phenolic index, and total contents of condensed tannins and anthocyanins of *Fondillón* wines as affected by the "solera" factor.

[†] ** and *** significant at *p* < 0.01, and 0.001, respectively. [‡] Values (mean of three replications) followed by the same letter, within the same row, were not significantly different (*p* < 0.05), according to Tukey's least significant difference test. [¶] Y mean yellow color, *R* mean red color, and *B* mean blue color.

Considering all the above, it can be concluded that the DPPH[•] method seemed the most suitable one to study the antioxidant activity of *Fondillón*, and that the antioxidant activity in this type of wine was clearly linked to the total anthocyanins content.

Another important conclusion of this section was that the *Fondillón solera* 1960 was the most special sample because it had the highest contents of phenolic compounds, condensed tannins, and anthocyanins, which led to (i) the highest values of antioxidant activity (all three methods under study, ABTS^{•+}, FRAP, and DPPH[•]), and (ii) the highest values of color density and intensity, as defined by intense red and blue colors.

3.3. Volatile Compounds

The HS-SPME-GC-MS analysis identified a total of 56 volatile compounds in all the analyzed wines, among which 34 showed significant differences (p < 0.05). The chemical profile consisted of 10 chemical families: 27 esters, 11 alcohols, 4 aldehydes, 4 alkanes, 3 ketones, 3 norisoprenoids, 1 organic acid, 1 terpene, 1 dioxolane, and 1 acetal, with esters being the predominant chemical group contributing to *Fondillón* odor and aroma (Table S3). No clear trends were found for the effect of the "*solera*" factor on the content of volatile compounds; thus, general comments will be presented in this section.

Esters are aromatic compounds providing and enhancing fruit aromas in wines; in general, the most important ones are ethyl fatty acid esters and acetates [38]. Diethyl butanedioate, ethyl octanoate, and ethyl acetate were the most abundant esters in the nine *Fondillón* samples under study and contributed to their red berries fragrance (Table 3).

Other NUMA number number 1 Acadablydyc NS 0.3 0.2 0.2 0.01 0.01 0.02 0.01 0.02 0.01 0.01 0.02 0.01 0.02 0.01 0.01 0.02 0.01 0.02 0.01 0.01 0.02 0.02 0.01 0.00 0.02 0.02 0.01 0.00 0.02 0.02 0.02 0.01 0.00 0.02 <t< th=""><th></th><th></th><th></th><th>1930</th><th>1944</th><th>1950</th><th>1960</th><th>1969</th><th>1975</th><th>1980</th><th>1987</th><th>1996</th></t<>				1930	1944	1950	1960	1969	1975	1980	1987	1996
Actication N 0.00 0.00 0.01 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01	Code	Volatile Compounds	ANOVA [†]	mg L ⁻¹								
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$			-	Tukey Multiple Range Test [‡]								
2 Bihand ** 10.3 b 10.9 ab 11.4 b 6.89 ab 5.66 b 90 ab 8.10 ab 1 Legrangi Jackhel NS 0.03 0.03 0.01 b 0.02 b 0.03 b <t< td=""><td>1</td><td>Acetaldehyde</td><td>NS</td><td>0.03</td><td>0.02</td><td>0.02</td><td>0.02</td><td>0.01</td><td>0.02</td><td>0.01</td><td>0.02</td><td>0.02</td></t<>	1	Acetaldehyde	NS	0.03	0.02	0.02	0.02	0.01	0.02	0.01	0.02	0.02
3 legregyl alcohol NS 0.02 0.01	2	Ethanol	**	12.3 a	10.9 ab	10.9 ab	11.4 a	6.98 ab	8.82 ab	5.66 b	9.09 ab	8.10 ab
4 Accit: acid NS 0.33 0.34 0.30 0.37 0.25 0.23 0.28 0.27 Hearing *** 0.05 0.02b 0.01b	3	Isopropyl alcohol	NS	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Hexare *** 0.15 0.01b 0.02b 0.01b 0.02b 0.01b 0.02b 0.01b 0.02b 0	4	Acetic acid	NS	0.33	0.34	0.30	0.37	0.26	0.23	0.28	0.32	0.27
6 Ebryl acetale ** 6.38 6.03 ab 5.16 b 6.09 ab 3.34 bc 3.36 c 3.66 c 4.51 bc 3.69 bc 7 Isolary Jackhal ** 0.08 a 0.01 ab 0.01 ab 0.03 abc 0.03 abc 0.03 bc 0.00 bc	5	Hexane	***	0.15 a	0.02 b	0.01 b	0.01 b	0.01 b	0.01 b	0.00 b	0.01 b	0.01 b
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	6	Ethyl acetate	**	6.38 a	6.03 ab	5.16 b	6.09 ab	3.84 bc	3.66 c	3.66 c	4.51 bc	3.69 bc
8 3-Methyl betanal NS 0.04 0.03 0.03 0.02 0.02 0.01 0.03 0.03 9 Bity propinate *** 1.04 1.02 kb 0.11 bc 0.10 bc 0.06 cc 0.07 bc 0.07 bc 10 2.4.5 trinedhyl -bitanad *** 1.24 ka 1.39 ka 1.39 ka 0.10 bc 0.06 cc 0.07 bc 0.07 bc 12 2.4.6 trinedhyl-bitanad ** 1.25 ka 1.25 ka 1.25 ka 1.25 ka 0.07 bc 0.07 bc </td <td>7</td> <td>Isobutyl alcohol</td> <td>**</td> <td>0.58 a</td> <td>0.44 abc</td> <td>0.44 abc</td> <td>0.49 ab</td> <td>0.33 abc</td> <td>0.34 abc</td> <td>0.23 c</td> <td>0.39 abc</td> <td>0.33 bc</td>	7	Isobutyl alcohol	**	0.58 a	0.44 abc	0.44 abc	0.49 ab	0.33 abc	0.34 abc	0.23 c	0.39 abc	0.33 bc
9 Ethyl propionale *** 0.18 a 0.11 be 0.12 be 0.10 be 0.10 be 0.00 be 0.09 be 0.07 be 11 Baamy Laichal ** 7.44 6.19 ab 5.38 be 4.46 be 3.04 ce 5.17 abc 4.57 Abc 13 Bahylischuryate ** 7.44 6.19 ab 5.38 be 4.46 be 3.04 ce 5.37 abc 4.57 Abc 13 Bahylischuryate ** 7.44 6.19 ab 5.38 be 4.04 be 0.05 be 0.04 be 0.05 be 1.55 be 0.16 be 0.02 be 0.12 be 1.55 be 0.55 be 0.03 be 0.04 be 0.01 be 0.02 be 0.01 be 0.02 be 0.01 be 0.02 b	8	3-Methyl butanal	NS	0.04	0.03	0.03	0.03	0.02	0.02	0.01	0.03	0.02
10 24,5 trimethyl-32-discolane *** 1,6,2,a 1,8,4,b 1,59,a 0,22,bc 1,03,4,bc 0,37,c 0,88,4,bc 0,77,c 0,78,4,bc 0,77,c 0,78,4,bc 0,77,c 0,78,4,bc 0,77,c 0,78,4,bc 0,77,c 0,78,4,bc 0,77,5,c 0,78,4,bc 0,77,5,c 0,78,4,bc 0,77,5,c 0,72,4,bc 0,77,c 0,78,4,c 0,03,c 0,	9	Ethyl propionate	***	0.18 a	0.11 bc	0.12 abc	0.14 ab	0.10 bc	0.10 bc	0.06 c	0.09 bc	0.07 bc
11 Learnyl alcohol ** 7.4 a 6.19 ab 5.38 ab 6.00 ab 398 bc 4.46 bc 3.01 c 5.17 abc 4.67 abc 12 EMPI labberyrate ** 0.13 0.10 bc 0.01 bc 0.02 0.01 bc 0.01 bc 0.02 0.02 bc 0.02 bc </td <td>10</td> <td>2,4,5-trimethyl-1,3-dioxolane</td> <td>***</td> <td>1.62 a</td> <td>1.38 ab</td> <td>1.48 ab</td> <td>1.59 a</td> <td>0.82 bc</td> <td>1.03 abc</td> <td>0.57 c</td> <td>0.98 abc</td> <td>0.78 bc</td>	10	2,4,5-trimethyl-1,3-dioxolane	***	1.62 a	1.38 ab	1.48 ab	1.59 a	0.82 bc	1.03 abc	0.57 c	0.98 abc	0.78 bc
12 2-Methyl-i-butando ** 2.93 ab 2.03 ab 2.00 ab 1.25 bc 1.08 bc 1.18 bc 1.13 bc 2.01 ab 0.01 ab 0.01 ab 0.01 ab 0.01 ab 0.01 ab 0.02 ab 0.07 bc 0.08 bc 0.01 ab 0.02 ab 0.01 ab 0.02 ab 0.02 ab 0.01 ab 0.02 ab 0.03 ab 0.02 ab 0.03 ab 0.02 ab 0.03 ab 0.02 ab 0.03 ab	11	Isoamyl alcohol	**	7.44 a	6.19 ab	5.38 abc	6.00 ab	3.98 bc	4.46 bc	3.04 c	5.17 abc	4.57 abc
13 Ethyl sockuty arctate ** 0.15 a 0.10 abc 0.11 ab 0.12 ab 0.07 bc 0.07 bc 0.01 bc 0.01 abc 0.08 bc 15 2.3-Butanediol NS 0.23 0.13 0.22 0.02 0.02 0.02 0.01 0.02 0.02 0.02 15 2.3-Butanediol NS 0.23 0.14 0.15 0.16	12	2-Methyl-1-butanol	**	2.93 a	2.33 ab	2.00 abc	2.22 abc	1.56 bc	1.68 bc	1.15 c	2.05 abc	1.72 bc
14 lobutyl actale NS 0.03 0.03 0.02 0.01 0.02 0.02 0.02 5 2.3-Butanchio NS 0.04 0.02 0.04 0.03 0.03 0.02 0.02 0.02 16 2.4-Butanchio NS 0.03 0.02 0.	13	Ethyl isobutyrate	**	0.15 a	0.10 abc	0.11 ab	0.12 ab	0.07 bc	0.08 bc	0.04 c	0.10 abc	0.08 bc
15 2.3-Brannekind NS 0.24 0.14 0.23 0.24 0.14 0.20 0.14 0.15 0.16 16 2.4-Bernande *** 0.18 ab 0.11 bc 0.02 0.01 0.02 0.02 0.01 0.02 0.02 0.01 0.02 0.02 0.01 0.02 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.01 0.02 0.03 0.02 0.03 0.02 0.03 0.02 0.03 0.02 0.03 0.02 0.03 0.02 0.03 0.02 0.03 0.02 0.02 0.01 <td< td=""><td>14</td><td>Isobutyl acetate</td><td>NS</td><td>0.03</td><td>0.03</td><td>0.02</td><td>0.03</td><td>0.02</td><td>0.01</td><td>0.02</td><td>0.02</td><td>0.02</td></td<>	14	Isobutyl acetate	NS	0.03	0.03	0.02	0.03	0.02	0.01	0.02	0.02	0.02
16 2-Hexanol NS 0.04 0.02 0.04 0.04 0.03 0.02 0.02 0.02 0.02 17 Ethyl lactate 0.98 ab 0.81 ab 0.12 b 0.20 a 0.02 b 0.04 bc 0.44 bc 0.03 bc 0.04 bc	15	2,3-Butanediol	NS	0.22	0.19	0.23	0.24	0.14	0.20	0.14	0.15	0.16
17 Erbyl butanete *** 0.13 bb 0.12 bb 0.26 a 0.10 bb 0.07 bb 0.17 ab 0.12 bb 18 Ethyl butanete ** 0.27 abc 0.25 abc 0.25 abc 0.25 abc 0.46 bc 0.40 cc 0.47 bc 0.17 bc 19 Furfural ** 0.12 ab 0.17 bc 0.23 abc 0.07 bc 0.02 abc 0.01 bc 0.01 cc 0.03 abc 0.02 bc 0.07 bc 0.02 abc 0.01 bc 0.01 cc 0.02 abc 0.02 bc 0.03 bc 0.02	16	2-Hexanol	NS	0.04	0.02	0.04	0.04	0.03	0.03	0.02	0.02	0.02
18 Erbyl lactate *** 0.98 abc 0.88 abc 1.09 a 0.52 bc 0.60 bc 0.40 c 0.54 bc 0.47 bc 20 Erbyl 2-metrylbutyrate ** 0.12 a 0.09 abc 0.10 ab 0.09 abc 0.06 bc 0.07 abc 0.14 c 0.09 abc 0.07 bc 21 Erbyl sorvalerate ** 0.23 abc 0.21 abc 0.16 abc 0.16 abc 0.12 c 0.23 ab 0.21 abc 23 1-Hexanol ** 0.30 a 0.02 abc 0.16 bc 0.16 bc 0.12 c 0.23 ab 0.21 abc 24 I-Hexanol ** 0.30 a 0.02 abc 0.30 abc 0.03 abc 0.03 abc 0.03 abc 0.02 abc 0.16 bc 0.12 c 0.23 abc 0.16 abc 25 Fortynalexnor NS 0.03 abc 0.02 abc 0.03 abc 0.03 abc 0.03 abc 0.03 abc 0.02 abc 0.14 abc 0.24 abc	17	Ethyl butanoate	***	0.18 ab	0.13 b	0.12 b	0.26 a	0.10 b	0.09 b	0.07 b	0.17 ab	0.12 b
19 Furfural ** 0.27 abc 0.28 abc 0.17 bc 0.02.2 abc 0.11 c 0.019 bc 0.07 bc 21 Ethyl isovalarite ** 0.02 ab 0.21 abc 0.23 ab 0.016 bc 0.07 bc 0.02 ab 0.016 bc 0.02 ab 0.016 bc 0.02 ab 0.016 bc 0.02 ab 0.03 bc 0.02 bc 0.02 bc 0.03 bc	18	Ethyl lactate	***	0.98 ab	0.80 abc	0.84 abc	1.09 a	0.52 bc	0.60 bc	0.40 c	0.54 bc	0.48 c
20 Ehyl 2-methylbutyrate ** 0.12 ab 0.09 abc 0.06 bc 0.07 abc 0.04 c 0.09 abc 0.017 bbc 21 Ehyl 2-methylbutyrate ** 0.32 a 0.21 abc 0.23 abc 0.16 bc 0.16 bc 0.12 c 0.23 ab 0.21 abc 22 Isamyl acetate ** 0.63 a 0.017 bc 0.23 bb 0.016 bc 0.16 bc 0.16 bc 0.12 c 0.23 ab 0.21 abc 0.34 bb 0.24 bb 0.36 bb 0.02 bb 0.04 bb 0.02 bb 0.04 bb 0.05 bb 0.03 c 0.01 bb 0.02 bb	19	Furfural	**	0.27 abc	0.25 abc	0.28 ab	0.35 a	0.17 bc	0.22 abc	0.11 c	0.19 bc	0.17 bc
21 Effyd isoxulerate ** 0.25 a 0.21 abc 0.23 ab 0.12 bc 0.16 abc 0.10 c 0.22 ab 0.16 abc 22 1-Hexanol ** 0.63 a 0.22 ab 0.17 bc 0.23 ab 0.27 ab 0.27 ab 0.22 bb 0.34 bb 0.34 bb 0.34 bb 23 Iscamyl acetate ** 0.62 a 0.57 bb 0.52 ab 0.02 ab 0.02 ab 0.03 ab 0.02 ab 0.03 ab 0.02 ab 0.02 ab 0.03 ab 0.02 ab 0.03 ab 0.02 ab 0.03 ab 0.03 ab 0.03 ab 0.03 ab 0.04 ab </td <td>20</td> <td>Ethyl 2-methylbutyrate</td> <td>**</td> <td>0.12 a</td> <td>0.09 ab</td> <td>0.10 ab</td> <td>0.09 abc</td> <td>0.06 bc</td> <td>0.07 abc</td> <td>0.04 c</td> <td>0.09 abc</td> <td>0.07 bc</td>	20	Ethyl 2-methylbutyrate	**	0.12 a	0.09 ab	0.10 ab	0.09 abc	0.06 bc	0.07 abc	0.04 c	0.09 abc	0.07 bc
22 1-Hexanol *** 0.30 a 0.22 ab 0.01 bc 0.16 bc 0.16 bc 0.12 c 0.23 ab 0.21 abc 23 Issumyl acetate ** 0.62 a 0.51 ab 0.37 b 0.23 ab 0.01 0.02 0.01 0.02 0.01 0.02 0.02 24 Ethyl pentanoate NS 0.03 0.03 0.02 0.03 0.03 0.01 0.02 0.02 25 1.1-Dethoxy-3-methylbutane NS 0.03 0.33 ab 0.34 ab 0.34 b 0.34 b 0.35 27 Benzaldehyde *** 0.50 a 0.35 ab 0.26 ab 0.29 bc 0.14 c 0.28 bc 0.31 bc 28 Ethyl hexanoate NS 0.03 0.02 0.01 0.01 0.02 0.01 0.01 0.02 0.01 0.01 0.02 0.01 0.01 0.02 0.01 0.01 0.02 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01	21	Ethyl isovalerate	**	0.25 a	0.21 abc	0.21 abc	0.23 ab	0.12 bc	0.16 abc	0.10 c	0.22 ab	0.16 abc
23 Istamyl acetate ** 0.62 0.37 0.29 0.36 0.43 0.24 0.43 0.24 0.43 0.24 0.24 0.03 0.02 0.02 0.03 0.02 0.02 0.01 0.02 0.02 0.02 25 Butyrolactone NS 0.03 0.03 0.02 0.03 0.03 0.01 0.02 0.02 27 Benzaldehyde *** 0.50 0.36 0.36 0.26 0.28 abc 0.24 bc	22	1-Hexanol	***	0.30 a	0.22 ab	0.17 bc	0.20 abc	0.16 bc	0.16 bc	0.12 c	0.23 ab	0.21 abc
24 Ethyl pertamonie NS 0.03 0.02 0.03 0.01 0.02 0.01 0.02 0.02 25 Bityrolactore NS 0.07 0.04 0.05 0.03 0.03 0.01 0.02 0.02 26 1.1-Dicthoxy-3-methylbutane NS 0.07 0.04 0.05 0.03 0.03 0.01 0.03 0.02 27 Bernzaldehyde *** 1.35 ab 1.08 abc 1.05 abc 1.44 a 0.66 c 0.82 abc 0.52 c 0.89 abc 0.74 bc 28 Ethyl hexanoate ** 1.35 ab 1.08 abc 1.05 abc 1.44 a 0.66 c 0.82 abc 0.52 c 0.89 abc 0.74 bc 29 Hexyl acetate NS 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.02 0.01	23	Isoamyl acetate	**	0.62 a	0.51 ab	0.37 b	0.52 ab	0.37 ab	0.29 b	0.36 b	0.43 ab	0.34 b
25 Butyrolactore NS 0.03 0.03 0.02 0.03 0.02 0.01 0.02 0.02 26 1,1 bethoys-methylbutane *** 0.50 a 0.35 ab 0.36 ab 0.03 ab 0.02 0.01 0.01 0.02 0.02 27 Benzyl alcohal *** 0.50 a 0.35 ab 0.36 ab 0.36 ab 0.29 bc 0.29 bc 0.14 c 0.28 bc 0.74 bc 28 Ethyl hexanote ** 0.03 0.02 0.02 0.01 0.01 0.01 0.02 0.01 01 Limonene NS 0.03 0.02 0.02 0.01 <td>24</td> <td>Ethyl pentanoate</td> <td>NS</td> <td>0.03</td> <td>0.02</td> <td>0.02</td> <td>0.03</td> <td>0.01</td> <td>0.02</td> <td>0.01</td> <td>0.02</td> <td>0.02</td>	24	Ethyl pentanoate	NS	0.03	0.02	0.02	0.03	0.01	0.02	0.01	0.02	0.02
26 1,1-Dicthory-3-methylbutane NS 0.07 0.04 0.05 0.05 0.03 0.01 0.03 0.02 27 Berazlalchylde *** 1.53 ab 1.08 abc 1.05 abc 1.44 a 0.66 c 0.82 abc 0.52 c 0.89 abc 0.74 bc 28 Ethyl hexanoate ** 1.35 ab 1.08 abc 1.05 abc 1.44 a 0.66 c 0.82 abc 0.52 c 0.89 abc 0.74 bc 29 Hexyl acetate NS 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.02 0.01	25	Butyrolactone	NS	0.03	0.03	0.02	0.03	0.02	0.02	0.01	0.02	0.02
27 Benzaldehyde *** 0.50 a 0.33 ab 0.36 ab 0.26 ab 0.22 bc 0.14 c 0.28 bc 0.31 bc 28<	26	1,1-Diethoxy-3-methylbutane	NS	0.07	0.04	0.05	0.05	0.03	0.03	0.01	0.03	0.02
28 Ethyl nexanoate **** 1.36 abc 1.06 abc 1.44 a 0.66 c 0.82 abc 0.32 c 0.08 abc 0.74 bc 30 Limonene NS 0.01 0.01 0.02 0.01 0.01 0.01 0.02 0.00 31 Berxyl alcohol NS 0.02 0.01 0.03 <	27	Benzaldehyde	***	0.50 a	0.35 ab	0.36 ab	0.36 ab	0.29 bc	0.29 bc	0.14 c	0.28 bc	0.31 bc
29 Hexyl acetate NS 0.03 0.02 0.02 0.02 0.01 0.01 0.01 0.02 0.01 30 Limonene NS 0.02 0.02 0.02 0.01 <	28	Ethyl hexanoate	**	1.35 ab	1.08 abc	1.05 abc	1.44 a	0.66 c	0.82 abc	0.52 c	0.89 abc	0.74 bc
31 Encryal alcohol NS 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.02 0.00 32 Ethyl 2-brexenoate NS 0.02 0.01 <td< td=""><td>29</td><td>Hexyl acetate</td><td>NS</td><td>0.03</td><td>0.02</td><td>0.02</td><td>0.02</td><td>0.01</td><td>0.01</td><td>0.01</td><td>0.02</td><td>0.01</td></td<>	29	Hexyl acetate	NS	0.03	0.02	0.02	0.02	0.01	0.01	0.01	0.02	0.01
31 Benzyl alcohol NS 0.02 0.02 0.01	30	Limonene	NS	0.01	0.01	0.01	0.02	0.01	0.01	0.01	0.02	0.00
32 Ethyl E-2-hexenoate NS 0.02 0.01	31	Benzyl alcohol	NS	0.02	0.02	0.02	0.01	0.01	0.02	0.01	0.01	0.02
33 Ethyl 2-turoate NS 0.01 <td>32</td> <td>Ethyl E-2-hexenoate</td> <td>NS NG</td> <td>0.02</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td>	32	Ethyl E-2-hexenoate	NS NG	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
34 Isoamy butyrate NS 0.02 0.01	33	Ethyl 2-turoate	NS NG	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
35 2-Nonanone NS 0.02 0.01	34	Isoamyl butyrate	NS	0.02	0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.01
36 5-Nonanome NS 0.01 <	35	2-Nonanoi	INS NG	0.02	0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.01
37 2-Nonlarone NS 0.05 0.02 0.03 0.02 0.02 0.01 0.01 0.02 0.01 0.01 0.02 0.02 38 Ethyl heptanoate NS 0.06 0.06 0.05 0.03 0.04 0.01 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.03 0.04 0.04 0.04 0.03 0.04 0.04 0.03 0.04 0.06 bc 0.06 bc 0.06 bc 0.07 bc 0.04 0.06 bc 0.06 <td>36</td> <td>3-Nonanone</td> <td>INS NG</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td> <td>0.01</td>	36	3-Nonanone	INS NG	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
39 Enry ineplandate NS 0.00 0.00 0.03 0.03 0.03 0.04 0.02 0.04 0.03 39 Phenethyl alcohol ** 0.09 ab 0.09 bc 0.10 a 0.06 abc 0.07 abc 0.04 c 0.06 abc 0.06 abc 40 Ethyl benzoate ** 0.09 ab 0.09 bc 0.10 a 0.06 abc 0.07 abc 0.04 c 0.06 abc 0.06 abc 41 Diethyl butnedioate ** 8.19 a 7.64 a 7.34 a 8.36 a 4.57 ab 5.68 ab 3.14 b 2.30 ab 4.64 ab 42 Ethyl phenyl acetate *** 0.10 ab 0.08 abc 0.05 bc 0.12 a 0.07 abc 0.04 c 0.05 abc 0.05 ab 43 Ethyl phenyl acetate *** 0.10 ab 0.08 abc 0.05 bc 0.03 a 0.04 c 0.05 bc 0.03 cd 0.04 bc 0.05 bc 0.05 c 0.03 cd 0.04 bc 0.05 abc 0.05 abc 0.05 abc 0.02 ab 0.01 ab 0.02 ab 0.02 ab 0.01 ab 0.02 ab 0.02 ab 0.02 ab 0.03 cd 0.04 bc<	3/	Z-INORATIONE Ethyd hantanaata	INS NC	0.05	0.05	0.02	0.05	0.02	0.02	0.01	0.03	0.02
30 Intentiny factorial 4.24 3.09 abc 5.09 abc 2.00 abc 1.00 abc 1.00 abc 1.00 abc 2.00 abc 1.00 abc 1.00 abc 2.00 abc 1.00 abc 0.06 abc 0.07 abc 0.04 c 0.06 abc 0.06 abc 0.06 abc 0.07 abc 0.04 c 0.06 abc 0.06 abc 0.07 abc 0.04 c 0.06 abc 0.06 abc 0.07 abc 0.04 c 0.06 abc 0.06 abc 0.07 abc 0.05 bc 0.03 c 0.12 a 0.01 ab 42 Ethyl optinosate *** 0.10 ab 0.08 abc 0.05 bc 0.05 bc 0.03 c 0.02 a 0.01 ab 0.06 abc 0.03 cd 0.04 c 0.06 abc 0.02 d 43 Ethyl splatcate *** 0.03 bcd 0.06 abc 0.01 a 0.07 ab 0.03 cd 0.04 c 0.03 abc 0.02 abc	20	Phopothyl alcohol	**	4.24 2	2.84 ab	2.09 abc	2.46 abc	2.25 hc	2.65 abc	1.78 c	2 15 abc	2.00 abc
41 Diefhyl butanedioate ** 8.19 a 7.64 a 7.34 a 8.36 a 4.55 ab 5.66 ab 3.14 b 5.00 ab 4.64 ab 42 Ethyl locatnoate *** 7.45 a 5.55 abc 6.67 ab 5.23 abc 3.40 c 4.77 abc 2.72 c 4.88 abc 4.25 bc 43 Ethyl locatnoate *** 0.10 ab 0.08 abc 0.07 abc 0.06 abc 0.07 abc 0.07 abc 0.07 abc 0.07 abc 0.07 abc 0.07 abc 0.06 abc 0.07 abc	40	Ethyl henzoate	**	4.24 a	0.09 ab	0.05 bc	0.10 a	2.55 bc	2.05 abc	0.04 c	0.06 abc	2.90 abc
42 Ehrlyl octamoate *** 7.45 a 5.57 abc 6.67 abc 5.23 abc 3.40 c 4.77 abc 2.72 c 4.88 abc 4.25 bc 43 Ethyl octamoate *** 0.10 ab 0.03 bc 0.05 bc 0.03 c 0.12 a 0.010 ab 44 Phenethyl acetate *** 0.00 ba 0.06 bc 0.03 cd 0.07 ab 0.03 cd 0.04 c 0.05 bc 0.03 cd 0.04 abc 0.05 bc 0.02 abc 0.04 c 0.05 bc 0.03 cd 0.04 abc 0.05 bc 0.03 cd 0.04 bcd 0.02 db 0.02 db 45 Ethyl gitatrate *** 0.05 00 abc 0.04 abc 0.01 ab 0.07 ab 0.03 cd 0.04 bcd 0.02 db 0.01 ab 46 Ethyl gitatrate NS 0.03 abc 0.07 ab 0.02 c 0.01 bbc 0.16 abc 0.11 bc 0.16 abc 0.11 bc 0.10 bbc 0.16 abc 0.11 bc 0.10 bbc 0.02 c 0.02 c 0.02 c 0.02 c 0.02 c <td>41</td> <td>Diethyl butanedioate</td> <td>**</td> <td>8 19 2</td> <td>764 a</td> <td>7 34 a</td> <td>8 36 3</td> <td>4 55 ab</td> <td>5.68 ab</td> <td>3.14 b</td> <td>5.30 ab</td> <td>4.64 ab</td>	41	Diethyl butanedioate	**	8 19 2	764 a	7 34 a	8 36 3	4 55 ab	5.68 ab	3.14 b	5.30 ab	4.64 ab
43 Ethylphenyl acetate *** 0.10 ab 0.08 abc 0.07 abc 0.07 abc 0.03 bc 0.03 bc 0.01 abc 0.07 abc 0.03 bc 0.02 bd 0.02 0.02 0.02 0.02 0.01 0.02 0.01 46 Ethyl spirare *** 0.03 a 0.02 0.02 0.02 0.02 0.02 0.02 0.03 bc 0.04 bc 0.04 bc	42	Ethyl octanoate	***	7.45 a	5 55 abc	6.67 ab	5.23 abc	3.40 c	4 77 abc	2 72 c	4.88 abc	4.04 ab
44 Phenethyl acetate ** 0.08 a 0.07 ab 0.06 abc 0.04 c 0.05 bc 0.06 abc 0.05 abc 45 Ethyl salicylate *** 0.05 do 0.06 abc 0.07 ab 0.03 cd 0.07 ab 0.03 cd 0.04 bc 0.02 abc 46 Ethyl salicylate NS 0.03 0.02 0.02 0.02 0.02 0.02 0.01 bc 0.16 abc 0.16 abc 0.04 bc 0.02 d 0.01 47 Vitispirane *** 0.07 a 0.05 abc 0.08 bc 0.03 bc 0.08 c 0.11 bc 0.10 bc 0.16 ab 0.11 bc 48 Ethyl nonanoate ** 0.02 a 0.02 b 0.01 ab 0.05 abc 0.03 abc 0.03 ab 0.01 ab 0.02 ab 0.03 ab 0.01 ab 0.05 abc 0.03 ab 0.01 ab 0.05 abc 0.03 ab 0.01 ab 0.01 ab 0.01 ab 0.02 ab<	43	Ethylphenyl acetate	***	0.10 ab	0.08 abc	0.05 hc	0.12 a	0.07 abc	0.05 bc	0.03 c	0.12 a	0.10 ab
15 Ethyl saftyrlate *** 0.05 bcd 0.06 abc 0.07 ab 0.03 cd 0.02 ab 0.02 ab 0.02 db 0.03 cc 0.03 cc	44	Phenethyl acetate	**	0.08 a	0.07 ab	0.05 bc	0.08 a	0.06 abc	0.04 c	0.05 bc	0.06 abc	0.05 abc
46 Ethyl glutarate NS 0.03 0.02 0.02 0.02 0.02 0.01 0.02 0.01 47 Vitispirane *** 0.02 0.15 ab 0.12 bc 0.10 bc 0.08 c 0.11 bc 0.10 bc 0.16 ab 0.11 bc 48 Ethyl nanoate ** 0.07 a 0.05 abc 0.07 b0 bc 0.08 cc 0.11 bc 0.03 cb 0.06 abc 0.03 bc 0.03 cb 0.04 bc 0.04 bc 49 Tridecane NS 0.03 0.01 0.01 0.01 0.01 0.00 0.01 0.01 0.01 0.00 0.01 0.01 0.01 0.01 0.00 0.01 <	45	Ethyl salicylate	***	0.05 bcd	0.06 abc	0.10 a	0.07 ab	0.03 cd	0.07 ab	0.03 cd	0.04 bcd	0.02 d
47 Vitispirame *** 0.20 a 0.15 ab 0.12 bc 0.10 bc 0.08 c 0.11 bc 0.10 bc 0.16 ab 0.11 bc 48 Ethyl nonanoate ** 0.07 a 0.05 abc 0.07 ab 0.06 abc 0.03 bc 0.03 bc 0.03 bc 0.03 abc 0.07 ab 0.06 abc 0.03 bc 0.03 c 0.05 abc 0.04 bc 49 Tridecane NS 0.03 0.01 0.03 0.02 0.03 c 0.03 c <td>46</td> <td>Ethyl glutarate</td> <td>NS</td> <td>0.03</td> <td>0.02</td> <td>0.02</td> <td>0.02</td> <td>0.02</td> <td>0.02</td> <td>0.01</td> <td>0.02</td> <td>0.01</td>	46	Ethyl glutarate	NS	0.03	0.02	0.02	0.02	0.02	0.02	0.01	0.02	0.01
48 Ethyl nonanoate ** 0.07 a 0.05 abc 0.07 ab 0.06 abc 0.03 bc 0.03 abc 0.03 cb 0.04 bc 49 Tridecane NS 0.03 0.01	47	Vitispirane	***	0.20 a	0.15 ab	0.12 bc	0.10 bc	0.08 c	0.11 bc	0.10 bc	0.16 ab	0.11 bc
49 Tridecane NS 0.03 0.01 <t< td=""><td>48</td><td>Ethyl nonanoate</td><td>**</td><td>0.07 a</td><td>0.05 abc</td><td>0.07 ab</td><td>0.06 abc</td><td>0.03 bc</td><td>0.05 abc</td><td>0.03 c</td><td>0.05 abc</td><td>0.04 bc</td></t<>	48	Ethyl nonanoate	**	0.07 a	0.05 abc	0.07 ab	0.06 abc	0.03 bc	0.05 abc	0.03 c	0.05 abc	0.04 bc
50 TDN *** 0.19 a 0.12 b 0.19 bc 0.06 c 0.09 bc 0.07 c 0.13 ab 0.08 bc 51 Ethyl decanoate *** 2.12 ab 1.82 abc 2.83 a 1.82 abc 1.70 ab 0.09 bc 0.06 c 0.07 ab 0.83 c 1.52 bc 1.46 bc 52 Tetradecane ** 0.08 a 0.01 b 0.01 b 0.00 b 0.01 b 0.01 b 0.01 b 0.01 b 0.00 b 0.01 b 0.01 b 0.01 b 0.01 b 0.00 b 0.01 b 0.00 b 0.00 b 0.00 b 0.00 b 0.01 b 0.02 b 0.01 b 0.00 b	49	Tridecane	NS	0.03	0.01	0.01	0.01	0.01	0.01	0.00	0.01	0.01
51 Ethyl decanoate *** 2.12 ab 1.82 abc 2.83 a 1.82 abc 1.17 bc 1.70 abc 0.83 c 1.52 bc 1.46 bc 52 Tetradecane ** 0.08 a 0.01 b 0.01 b 0.00 b 0.0	50	TDN	***	0.19 a	0.12 b	0.13 b	0.09 bc	0.06 c	0.09 bc	0.07 c	0.13 ab	0.08 bc
52 Tetradecane ** 0.08 a 0.01 b 0.01 b 0.00 b 0.00 b 0.00 b 0.01 b 0.01 b 53 Ethyl 3-methylbutyl butanedioate ** 0.11 a 0.09 ab 0.08 ab 0.10 ab 0.06 b 0.07 ab 0.04 c 0.06 b 0.06 b 0.06 b 0.00 b 0.01 b 54 Isoamyl octanoate *** 0.11 a 0.02 b 0.01 b 0.00 b 0.01 b 0.00 c 0.06 b 0.07 ab 0.04 c 0.06 b 0.06 b 55 Pentadecane *** 0.11 a 0.02 b 0.01 b 0.00 b	51	Ethyl decanoate	***	2.12 ab	1.82 abc	2.83 a	1.82 abc	1.17 bc	1.70 abc	0.83 c	1.52 bc	1.46 bc
53 Ethyl 3-methylbutyl butanedioate ** 0.11 a 0.09 ab 0.08 ab 0.10 ab 0.06 b 0.07 ab 0.04 c 0.06 b 0.06 b 0.06 cb 54 Isoamyl octanoate NS 0.01 0.00 0.01 0.00 0.01 0.00 b	52	Tetradecane	**	0.08 a	0.01 b	0.01 b	0.01 b	0.00 b	0.01 b	0.00 b	0.01 b	0.01 b
54 Isoamyl octanoate NS 0.01 0.00 0.01 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.00 0.01 0.01 0.00 0.01 0.00 0.01 0.01 0.00 0.00 0.001 0.01 0.00 0.00 0.001 0.01 0.00 0.001 0.00 0.001 0.01 0.00 0.001 <td>53</td> <td>Ethyl 3-methylbutyl butanedioate</td> <td>**</td> <td>0.11 a</td> <td>0.09 ab</td> <td>0.08 ab</td> <td>0.10 ab</td> <td>0.06 b</td> <td>0.07 ab</td> <td>0.04 c</td> <td>0.06 b</td> <td>0.06 b</td>	53	Ethyl 3-methylbutyl butanedioate	**	0.11 a	0.09 ab	0.08 ab	0.10 ab	0.06 b	0.07 ab	0.04 c	0.06 b	0.06 b
55 Pentadecane *** 0.11 a 0.02 b 0.01 b 0.00 b <td>54</td> <td>Isoamyl octanoate</td> <td>NS</td> <td>0.01</td> <td>0.00</td> <td>0.01</td> <td>0.01</td> <td>0.00</td> <td>0.01</td> <td>0.00</td> <td>0.00</td> <td>0.01</td>	54	Isoamyl octanoate	NS	0.01	0.00	0.01	0.01	0.00	0.01	0.00	0.00	0.01
56 2,3,5-Trimethylnaphthalene NS 0.03 0.03 0.04 0.03 0.02 0.02 0.02 0.03 0.03	55	Pentadecane	***	0.11 a	0.02 b	0.01 b	0.00 b	0.00 b	0.00 b	0.00 b	0.00 b	0.00 b
	56	2,3,5-Trimethylnaphthalene	NS	0.03	0.03	0.04	0.03	0.02	0.02	0.02	0.03	0.03

⁺ NS = not significant at p < 0.05; **, ***, significant at p < 0.01, and 0.001, respectively. [‡] Values (mean of three replications) followed by the same letter, within the same row, were not significantly different (p < 0.05), according to Tukey's least significant difference test.

In the same way, alcohols played an important role in the wines under analysis; they can come from the grapes themselves or from the yeast metabolism [38]. In *Fondillón*, the predominant ones were isoamyl alcohol, 2-methyl-1-butanol, and phenethyl alcohol and were responsible for fruity, floral, and honey notes.

The norisoprenoids are key compounds in the wine aroma profile and belong to the carotenoid metabolism. During wine aging, glycosidically-bound aroma compounds coming from the carotenoid metabolism can be subjected to slow acid hydrolysis rendering norisoprenoids such as 1,1,6-trimethyl-l,2-dihydronaphthalene (TDN) and vitispirane isomers [38,39]. TDN has been reported as the compound responsible for the kerosene bottle-aged character of Riesling wines [38,40], while vitispirane contributes to nut flavor notes [41]. There was no significant effect of the "*solera*" factor on the contents of (i) TDN (range 0.06 and 0.19 mg L⁻¹, with a mean of 0.13 mg L⁻¹) and (ii) vitispirane (range 0.08 and 0.20 mg L⁻¹, with a mean of 0.14 mg L⁻¹).

Regarding the *Fondillón solera* 1960 (the sample identified as the one having the highest antioxidant activity and highest contents of bioactive compounds, such as phenolic compounds), it contained high contents of key aroma compounds (ethyl butanoate, ethyl lactate, furfural, ethyl hexanoate, ethyl benzoate, etc.) (Table 3), which contributed to intense fruity, nutty, caramel, sweet, and floral notes.

3.4. Descriptive Sensory Analysis with Trained Panel

According to the legislation, *Fondillón* is defined as a wine being sweet, balanced, with good structure, and having intense aroma with predominance of ripe fruit, nuts, well-integrated wood and toasted coffee, and long aftertaste [23]. The descriptive profiles of the nine *Fondillón* samples (66 years of difference in the aging time) under analysis only showed statistically significant differences for 10 of the 22 attributes, which were used to evaluate the quality of these wines (Figure S1); these attributes were: alcohol (o, f), fruity (o, f), spicy (o, f), sweetness, sourness, astringency, and aftertaste (Table 4).

The attribute "aftertaste" is defined as a pleasant sensation, and it is considered by most of the experts and consumers as one of the most relevant; besides, it was significantly affected by the "*solera*" factor (Table 4). It was found that the sample with the longest aftertaste was that of the *solera* 1960. While the samples of *soleras* 1950 and 1987 were on the opposite side (shortest aftertaste, indicating perhaps a less complex organoleptic profile. Positive and significant correlations were found among aftertaste and (i) sweetness ($R^2 = 0.462$; p < 0.05) and (ii) fruity flavor ($R^2 = 0.467$; p < 0.05).

Table 4. Descriptive sensory analysis of wines aged in Fondillón wines as affected by the "solera" factor.

A 11		Fondillón solera										
Attribute	ANOVA [†]	1930	1944	1950	1960	1969	1975	1980	1987	1996		
			Tukey Multiple Range Test ‡									
Appearance												
Color	NS	5.2	5.2	5.2	5.0	5.2	5.7	5.8	5.3	5.7		
Odor												
Alcohol	*	6.0 a	5.0 ab	3.7 b	4.5 ab	4.2 ab	4.5 ab	5.0 ab	4.0 ab	3.7 b		
Fruity	**	7.0 a	5.2 ab	3.3 b	5.0 ab	4.7 ab	4.8 ab	5.2 ab	4.5 b	3.7 b		
Floral	NS	1.3	1.5	1.5	1.7	1.7	1.7	1.8	1.7	1.5		
Mediterranean forest	NS	2.0	1.8	2.0	1.8	1.5	1.8	1.7	1.7	1.7		
Spicy	*	4.2 a	3.5 ab	2.0 b	3.7 ab	3.3 ab	3.3 ab	3.8 a	3.5 ab	3.0 ab		
Animal	NS	1.5	2.2	1.0	1.7	1.7	2.2	2.0	1.7	1.3		
Toasted	NS	6.7	5.7	4.0	4.2	5.0	4.7	5.3	4.7	4.2		
Chemical	NS	0.8	2.5	2.5	2.7	2.3	2.7	3.5	2.0	1.3		
Basic taste												
Sweetness	*	3.0 ab	5.0 a	2.7 b	3.8 ab	3.7 ab	3.7 ab	3.2 ab	3.0 ab	2.7 b		
Sourness	**	4.5 a	3.5 abc	2.5 c	3.5 abc	3.0 bc	3.8 abc	4.2 ab	3.3 abc	2.8 bc		
Bitterness	NS	1.5	1.3	2.2	2.0	2.5	2.5	1.8	1.8	1.5		

A 11		Fondillón solera								
Attribute	ANOVA '	1930	1944	1950	1960	1969	1975	1980	1987	1996
					Tukey N	/ultiple Ran	ge Test ‡			
Flavor										
Alcohol	*	5.7 a	5.0 ab	4.0 b	5.2 ab	4.5 ab	4.7 ab	4.7 ab	4.5 ab	4.5 ab
Fruity	*	4.8 a	4.7 a	3.3 b	4.7 a	4.8 a	4.5 a	4.0 ab	4.0 ab	3.5 ab
Floral	NS	0.8	1.5	1.2	2.0	2.0	1.8	1.5	1.5	1.3
Mediterranean forest	NS	1.7	1.7	0.8	1.7	2.0	2.0	1.7	1.7	1.5
Spicy	*	3.8 a	3.0 ab	2.8 b	3.3 ab	3.0 ab	3.0 ab	3.2 ab	3.0 ab	2.8 b
Animal	NS	1.0	1.5	1.0	1.7	1.7	1.8	1.3	1.7	1.2
Toasted	NS	5.5	4.8	3.3	4.7	5.3	5.0	4.8	4.2	3.8
Chemical	NS	0.8	1.5	1.8	2.0	1.3	2.3	2.2	1.7	1.7
Astringency	**	1.0 b	0.8 b	0.8 b	0.8 b	1.2 b	2.0 a	1.2 b	1.2 b	1.0 b
Aftertaste	***	6.3 bc	6.8 ab	3.7 d	7.2 a	6.3 bc	6.3 bc	5.8 c	4.2 d	6.3 bc

Table 4. Cont.

⁺ NS = not significant at p < 0.05; *, **, *** significant at p < 0.05, 0.01, and 0.001, respectively. [‡] Values (mean of 8 trained panelists) followed by the same letter, within the same row, were not significantly different (p < 0.05), according to LSD least significant difference test.

3.5. Affective Sensory Analysis

The consumer profile was: 63% men (37% women), of which 5%, 18%, 45%, and 32% belonged to the age ranges 18–24, 25–39, 40–59, and 60–74%, respectively.

Samples of *soleras* 1944 (6.3), 1960 (6.2), and 1996 (6.1) obtained the highest overall liking values, followed by 1930 (5.8) and 1987 (5.8) samples (Table 5); these values led to an overall mean of ~6.0, which means that consumers liked the wines "slightly". This value can be taken as a high degree of satisfaction because consumers tend not to use the extreme values of the scale, concentrating their opinions in the middle part. On the other hand, *Fondillón* samples of *soleras* 1980, 1975, 1969, and 1950 showed the lowest liking values, with a mean of 5.1, which means that the wines were neither like nor dislike. Similar trends were found for the other two affective parameters under study, sweetness and aftertaste, with *Fondillón solera* 1960 having the highest liking scores.

When the effects of the gender and age factors were studied, it was surprisinly seen that young women, below 35 years old, were the ones with the highest liking score. This observation did not agree with the preconceived idea that the typical *Fondillón* consumer was a man of 40–50 years old, with high education and income. This surprising experimental observation opened a whole new perspective of research and revealed the important need to prepare a detailed study to determine which is the current profile of *Fondillón* and which are the options to promote *Fondillón* consumption among new consumers.

Figure 2a shows the preference percentage of the Spanish consumers for the *Fondillón* samples under study, and Figure 2b the reasons why they chose their preferred sample. The *Fondillón solera* 1960 was chosen as the most liked one (24%), mainly because of its color, its toasted (woody) odor and flavor, its sweetness, its toffee and coffee notes, and its long aftertaste. Besides, the wine obtaining the highest purchase intention (59%) was that of the *solera* 1960 (Figure 2c), with sample of *solera* 1980 being on the opposite side (30%).

_

	Overall	Sweetness	Aftertaste						
C 1	ANOVA ⁺								
Solera	***	***	***						
	Tukey Multiple Range Test ‡								
1930	5.8 b	5.1 bc	5.9 ab						
1944	6.3 a	5.8 ab	6.3 a						
1950	5.4 bc	5.1 bc	6.1 a						
1960	6.2 a	6.1 a	6.1 a						
1969	5.5 bc	5.1 bc	5.7 ab						
1975	5.2 bc	5.1 bc	5.6 ab						
1980	4.3 c	4.8 c	4.9 b						
1987	5.8 b	5.7 b	5.9 ab						
1996	6.1 ab	6.3 a	6.2 a						

Table 5. Affective sensory analysis of *Fondillón* wines as affected by the "solera" factor.

[†] *** significant at p < 0.001. [‡] Values (mean of 123 consumers) followed by the same letter, within the same column, were not significantly different (p < 0.05), according to LSD least significant difference test.





Figure 2. Consumer preference (**a**); main reasons to choose 1960 *Fondillón* as the favorite (**b**); and purchase intention (**c**).

4. Conclusions

The effect of the *solera* age on nine *Fondillón* samples was studied, by analyzing several quality, functional, and sensory parameters together with their consumer liking. In most of the cases, it seems that no clear trend was found for the "solera" factor perhaps due to the long aging time of the wines, above 25 years. Independently of the solera effect, the Fondillón solera 1960 was the most special one due to its high contents of phenolic compounds, condensed tannins, anthocyanins, high antioxidant activity, intense red and blue notes, and due to its high contents of key volatile compounds (e.g., ethyl butanoate and furfural), which provides the wine with nutty, caramel, and sweet notes. There was a positive correlation among overall liking with a long aftertaste and intense sweetness. The most important satisfaction and buying drivers for *Fondillón* consumers were color, toasted odor and flavor, sweetness, toffee and coffee notes, and long aftertaste. Additional conclusions were: (i) the DPPH[•] method showed the biggest differences among samples and can be considered as the most suitable one to study the antioxidant activity of *Fondillón*; and (ii) the Fondillón antioxidant activity showed a positive and significant correlation with the total anthocyanins content. The final recommendation based on the findings and conclusions of this study could be that Fondillón quality and consumer acceptance depend strongly on high contents of key compounds (e.g., anthocyanins) and high antioxidant capacity. Thus, Fondillón should be only prepared when the Monastrell grapes have high antioxidant potential besides proper total sugar content.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10 .3390/agronomy11091701/s1, Table S1: Affective questionnaire used in this study, Table S2: Volatile compounds, descriptors, and retention index of the *Fondillón* wines as affected by the "*solera*" factor [42,43], Table S3: Pearson's correlation coefficients (R) among basic enological parameters, chromatic characteristics, antioxidant activity (ABTS^{•+}, FRAP, DPPH[•]), total contents of condensed tannins (TCT) and anthocyanins (TA) and total polyphenol index (TPI) of the *Fondillón* wines as affected by the "*solera*" factor, Figure S1: Descriptive sensory profile of the *Fondillón* wines as affected by the "*solera*" factor (*, ** and *** mean p < 0.05, 0.01 and 0.001, respectively).

Author Contributions: Conceptualization, Á.A.C.-B. and D.L.-L.; methodology, H.I.-I.; software, H.I.-I. and L.L.; validation, Á.A.C.-B., F.H. and D.L.-L.; formal analysis, H.I.-I. and L.L.; investigation, H.I.-I., F.H. and L.L.; resources, D.L.-L. and F.H.; data curation, H.I.-I.; writing—original draft preparation, H.I.-I.; writing—review and editing, L.L.; visualization, F.H.; supervision, Á.A.C.-B., F.H. and D.L.-L. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: Authors want to truly thank Rafael Poveda and MGWines for providing the wines studied and by sharing all his deep knowledge on *Fondillón*.

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

References

- Post, W.M.; Kwon, K.C. Soil carbon sequestration and land-use change: Processes and potential. *Glob. Chang Biol.* 2000, 6, 317–327. [CrossRef]
- Agencia Estatal de Meteorología. Accumulated Precipitation (mm). Available online: https://www.aemet.es/es/ serviciosclimaticos/datosclimatologicos/valoresclimatologicos (accessed on 30 July 2021).
- 3. Escribano Francés, G.; Quevauviller, P.; San Martín González, E.; Vargas Amelin, E. Climate change policy and water resources in the EU and Spain. A closer look into the Water Framework Directive. *Environ. Sci. Policy* **2017**, *69*, 1–12. [CrossRef]
- 4. Sánchez-Bravo, P.; Edgar Chambers, V.; Noguera-Artiaga, L.; Sendra, E.; Edgar Chambers, I.V.; Carbonell-Barrachina, A.A. How consumers perceivewater sustainability (hydrosostainable) in food products and how to identify it by a logo. *Agronomy* **2020**, *10*, 1495. [CrossRef]

- Andreu-Coll, L.; Cano-Lamadrid, M.; Noguera-Artiaga, L.; Lipan, L.; Carbonell-Barrachina, Á.A.; Rocamora-Montiel, B.; Legua, P.; Hernández, F.; López-Lluch, D. Economic estimation of cactus pear production and its feasibility in Spain. *Trends. Food Sci. Technol.* 2020, 103, 379–385. [CrossRef]
- 6. Issa-Issa, H.; Noguera-Artiaga, L.; Sendra, E.; Pérez-López, A.J.; Burló, F.; Carbonell-Barrachina, A.A.; López-Lluch, D. Volatile Composition, Sensory Profile, and Consumers' Acceptance of *Fondillón. J. Food Qual.* **2019**, 2019, 5981762. [CrossRef]
- Council Regulation (EC) No 479/2008. Common Organisation of the Market in Wine. Official Journal of the European Union, 29 April 2008; No L 148/1-84.
- 8. Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Study of the formation mechanisms of some volatile compounds during the aging of sweet fortified wines. *J. Agric. Food Chem.* **1999**, 47, 2837–2846. [CrossRef] [PubMed]
- 9. Oliveira, C.M.; Ferreira, A.C.S.; De Freitas, V.; Silva, A.M.S. Oxidation mechanisms occurring in wines. *Food Res. Int.* **2011**, 44, 1115–1126. [CrossRef]
- 10. Pereira, V.; Albuquerque, F.; Cacho, J.; Marques, J.C. Polyphenols, antioxidant potential and color of fortified wines during accelerated ageing: The madeira wine case study. *Molecules* **2013**, *18*, 2997–3017. [CrossRef] [PubMed]
- 11. Gambuti, A.; Rinaldi, A.; Ugliano, M.; Moio, L. Evolution of phenolic compounds and astringency during aging of red wine: Effect of oxygen exposure before and after bottling. *J. Agric. Food Chem.* **2013**, *61*, 1618–1627. [CrossRef]
- 12. International Organization of Vine and Wine (OIV). *Compendium of International Methods of Wine and Must Analysis, Vol. 1*; OIV: Paris, France, 2020.
- 13. OIV (International Organization of Vine and Wine). *Compendium of International Methods of Wine and Must Analysis, Vol* 2; OIV: Paris, France, 2021.
- 14. Glories, Y. The color of red wines. Connaissance Vignevini 1984, 18, 253-271.
- Luna, J.M.; Garau, M.C.; Negre, A.; March, J.; Martorell, A. Composición Fenólica y Actividad Antioxidante de Variedades Minoritarias de Vid de las Islas Baleares. In Proceedings of the VII Foro Mundial del Vino, Logroño, La Rioja, Spain, 12–14 May 2010; Sáenz-Navajas, M.P., Fernández-Zurbano, P., Valentín, D., Ferreira-González, V., Eds.; Consejería de Agricultura, Ganadería y Desarrollo Rural: Logroño, La Rioja, Spain, 2010; p. 62.
- 16. Di Stefano, R.; Genfilini, N. Metodi per lo studio dei polifenoli dei vini. In *L'Enotecnico*; ResearchGate: Berlin, Germany, 1989; pp. 83–89.
- 17. Re, R.; Pellegrini, N.; Proteggente, A.; Pannala, A.; Yang, M.; Rice-Evans, C. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radic. Biol. Med.* **1999**, *26*, 1231–1237. [CrossRef]
- Katalinic, V.; Milos, M.; Kulisic, T.; Jukic, M. Screening of 70 medicinal plant extracts for antioxidant capacity and total phenols. Food Chem. 2006, 94, 550–557. [CrossRef]
- 19. Benzie, I.F.F.; Strain, J.J. The ferric reducing ability 7, of plasma (FRAP) as a measure of 'antioxidant power': The FRAP assay. *Anal. Biochem.* **1996**, 239, 70–76. [CrossRef]
- 20. Ribéreau-Gayon, P.; Stonestreet, E. Determination of anthocyanins in red wine. *Bulletin de la Societe Chimique de France* **1965**, *9*, 2649–2652. [PubMed]
- International Organization of Standardization. ISO 8586:2012. Sensory Analysis—General Guidelines for the Selection, Training and Monitoring of Selected Assessors and Expert Sensory Assessors. Available online: https://www.iso.org/standard/45352. html (accessed on 8 June 2021).
- 22. International Organization of Standardization. ISO/IEC 17065:2012. Conformity Assessment—Requirements for Bodies Certifying Products, Processes and Services. Available online: https://www.iso.org/standard/46568.html (accessed on 8 June 2021).
- 23. Orden 5/2011. Reglamento y Pliego de Condiciones de la Denominación de Origen Protegida Alicante y su Consejo Regulador. *Diario Oficial de la Comunitat Valenciana*, 16 November 2011; DOCV, No 6661/39267-39289.
- 24. Forino, M.; Picariello, L.; Rinaldi, A.; Moio, L.; Gambuti, A. How must pH affects the level of red wine phenols. *LWT* **2020**, *129*, 109546. [CrossRef]
- 25. Nogueira, J.M.F.; Nascimento, A.M.D. Analytical characterization of Madeira wine. J. Agric. Food Chem. 1999, 47, 566–575. [CrossRef] [PubMed]
- 26. Miranda, A.; Pereira, V.; Pontes, M.; Albuquerque, F.; Marques, J.C. Acetic acid and ethyl acetate in Madeira wines: Evolution with ageing and assessment of the odour rejection threshold. *Ciencia e Tecnica Vitivinicola* **2017**, *32*, 1–11. [CrossRef]
- 27. Gómez-Cebrián, R. Characterization of Fondillón Wines from the Denomination of Origin Alicante. Master's Thesis, Universidad Miguel Hernández de Elche, Elche, Alicante, Spain, 2015. (In Spanish).
- 28. Council Regulation (EEC) No 4252/88. Preparation and Marketing of Liquer Wines Produced in the Community. *Official Journal of the European Communities*, 21 December 1988; No L 373/59-65.
- González-Neves, g.; Balado, J.; Barreiro, L.; Bochicchio, R.; Gatto, G.; Gil, G.; Tessore, A.; Ferrer, M. Efecto de Algunas Prácticas de Manejo del Viñedo y de la Vinificación en la Composición Fenólica y el Color de los Vinos Tintos. In Proceedings of the X Congresso Brasileiro de Viticultura e Enologia, Anais, Bento Gonçalves, RS, Brazil, 3–5 December 2003.
- 30. Reyes-Cortés, C.A. Red Wine Quality usign Analysis of the Color Intensity and Anthocyanins Content (Degree Thesis, in Spanish); Universidad de Chile: Santiago de Chile, Chile, 2020.
- Gómez-Cordovés, C.; González-SanJosé, M.L. Interpretation of Color Variables during the Aging of Red Wines: Relationship with Families of Phenolic Compounds. J. Agric. Food Chem. 1995, 43, 557–561. [CrossRef]

- Del Fresno, J.M.; Morata, A.; Loira, I.; Escott, C.; Suárez Lepe, J.A. Evolution of the Phenolic Fraction and Aromatic Profile of Red Wines Aged in Oak Barrels. ACS Omega 2020, 5, 7235–7243. [CrossRef] [PubMed]
- Rivero-Pérez, M.D.; González-Sanjosé, M.L.; Muñiz, P.; Pérez-Magariño, S. Antioxidant profile of red-single variety wines microoxygenated before malolactic fermentation. *Food Chem.* 2008, 111, 1004–1011. [CrossRef]
- 34. Larrauri, J.A.; Sánchez-Moreno, C.; Rupérez, P.; Saura-Calixto, F. Free radical scavenging capacity in the aging of selected red spanish wines. *J. Agric. Food Chem.* **1999**, *47*, 1603–1606. [CrossRef]
- 35. Rivero-Pérez, M.D.; González-Sanjosé, M.L.; Ortega-Herás, M.; Muñiz, P. Antioxidant potential of single-variety red wines aged in the barrel and in the bottle. *Food Chem.* **2008**, *111*, 957–964. [CrossRef]
- Moreno, J.; Peinado, J.; Peinado, R.A. Antioxidant activity of musts from Pedro Ximénez grapes subjected to off-vine drying process. *Food Chem.* 2007, 104, 224–228. [CrossRef]
- López de Lerma, N.; Peinado, J.; Moreno, J.; Peinado, R.A. Antioxidant activity, browning and volatile Maillard compounds in Pedro Ximénez sweet wines under accelerated oxidative aging. LWT–Food Sci. Technol. 2010, 43, 1557–1563. [CrossRef]
- 38. Robinson, A.L.; Boss, P.K.; Solomon, P.S.; Trengove, R.D.; Heymann, H.; Ebeler, S.E. Origins of grape and wine aroma. Part 1. Chemical components and viticultural impacts. *Am. J. Enol. Vitic.* **2014**, *65*, 1–24. [CrossRef]
- 39. Full, G.; Winterhalter, P. Application of on-line coupled mass spectrometric techniques for the study of isomeric vitispiranes and their precursors of grapevine cv. Riesling. *Vitis* **1994**, *33*, 241–244.
- Winterhalter, P.; Sefton, M.A.; Williams, P.J. Volatile C13-Norisoprenoid Compounds in Riesling Wine Are Generated From Multiple Precursors. Am. J. Enol. Vitic. 1990, 41, 277–283.
- 41. Ángeles Pozo-Bayón, M.; Victoria Moreno-Arribas, M. Sherry wines. Adv. Food Nutr. Res. 2011, 63, 17–40. [CrossRef] [PubMed]
- 42. National Institute of Standards and Technology (NIST), NIST Chemistry Webbook. Available online: https://webbook.nist.gov/ chemistry (accessed on 8 July 2021).
- 43. Sigma-Aldrich. Flavors & Fragrances; Sigma-Aldrich: Saint Louis, MO, USA, 2012.