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Research Article

Volatile Composition, Sensory Profile, and Consumers' Acceptance of *Fondillón*

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No scientific information exists on quality attributes of *Fondillón*, a special naturally sweet wine produced from overripe Monastrell grapes and one of the only six wines that can use its own name according to European Union Regulations. The aim of this study was to analyze the composition (physicochemical and volatile profile) and sensory quality of this special wine. A specific lexicon to describe wines under the Alicante PDO was developed, using 28 attributes (11 flavor notes, 3 visual, 2 global, and 12 defects). Forty volatile compounds were isolated, and esters were the main chemical family of volatile compounds of *Fondillón* (~70%), followed by alcohols (~20%). Furthermore, two volatile compounds (TDN and vitispirane) were positively correlated with the age of the *Fondillón* samples, under the specific working conditions used in this study. According to a sensory study, this wine was appreciated by Spanish consumers as having intense fruity notes, high alcohol content, and some bitter and balsamic notes; however, further research is needed to identify the proper profile of *Fondillón* consumers and their buying and acceptance drivers.

1. Introduction

Wine fermentation turns grapes into wine. In this process, yeasts take natural sugars from mature grapes and convert them into alcohol and CO_2. Hence, most wines are dry or almost dry (they have no sweetness or residual sugar). However, we can find several wines produced through different processes that have some different amount of sugar. This sort of wines is called sweet or dessert wines. Along history, the amount of sugar in the finished wines was a key factor for conservation. Sweet wines were highly valued in ancient Rome and in the Middle Ages and were promoted and marketed within the Dutch and British wine trade of the early 18th century. However, nowadays, they represent a very small percentage of the global wine business. Nevertheless, there is a growing interest in high-quality sweet wines [1].

Dehydrating grapes can be reached in two ways, on-vine or off-vine [2]. Grapes can become overripe through several techniques, such as by exposure to sunlight (Passito, Pedro Ximenez, Málaga); by dehydration in closed rooms of hot or fresh air (Recioto, Vin Santo, Vin de Paille); by grape colonization by fungus *Botrytis cinerea*, causing noble rot (Alsace, Loire, Montbazillac, Sauternes, Tokaji, Trockenbeerenauslese); by leaving grapes to shrivel in the plot, where they may also be occasionally affected by noble rot (*Fondillón*, Spatlese, Tokaji Late harvest, Vendage Tardive); or by waiting until winter, causing grape dehydration by ice (Eiswein, Vi de Gel, ice wine) [3]. These

on-vine overripe grapes lead to naturally concentrated must, rich in sugars and volatile compounds. Natural sweet wines are mainly featured by their high sugar level, and their quality mainly depends on their aroma compounds [1]. However, still, little is known about the biochemistry behind this special sort of wine [2]. Wines produced according to this method are for example, Alsatia, Fiano, *Fondillón*, Jurançon, Pacherenc du Vic-Bilh, Picolit, Priorat rancid sweet, and Malvasia from La Palma and Lanzarote [4].

It is possible to classify sweet wines according to the winemaking process: fortified musts, fortified wines, and naturally sweet wines. Fortified musts (Muscat, mistelle) and fortified wines (Port wine, Sherry) are those in which fermentation is stopped by adding alcohol to the must or wine, respectively. On the other hand, naturally sweet wines, including *Fondillón*, come from overripe grapes and are nonfortified wines. These wines with a total alcoholic strength of not less than 15% by volume (abv), and an actual alcoholic strength of not less than 12% abv are produced without enrichment [5]. Owing to the high grape original sugar content, yeast metabolism implies high levels of alcohol (naturally above 15% abv); this high alcohol content is the most usual cause of cessation of fermentation in nonfortified dessert wines.

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]. *Fondillón* is a red wine produced in an oxidized (*rancio*) style from overripe Monastrell grapes; it is typically bottled and sold after a long aging period in oak barrel (minimum 10 years). *Fondillón* production was almost lost during the end of the 19th century, but fortunately, it was recovered around 1950; but, since then, no scientific approach has been done to fully characterize this wine and to promote its distinctive characteristics.

Consequently, the aim of this study was to develop a sensory protocol (mainly the lexicon) to properly describe the quality of the *Fondillón* wines being marketed in Spain. This is essential to guarantee that only those wines fulfilling the requirements of the PDO Alicante get the proper seal. To back up sensory data with instrumental and physicochemical data, the basic quality parameters and the volatile profile were also analyzed.

2. Materials and Methods

2.1. Wine Samples. Seven *Fondillón* samples (F1–F7) under the Alicante PDO were analyzed in this study, in triplicate (from different batches), to get the main characteristics of this type of wine (quality parameters, typical descriptive sensory profile, and volatile composition). During the first stage of the experiment, *Fondillón* samples were taken from the seven wineries in Alicante producing this product in 2015 and were kindly donated by the Regulatory Council of the Alicante PDO. Samples consisted of 3 commercial bottles (3 different batches) from each of the 7 wineries, with at least 10 years of aging, but some of the samples had up to 25 years.

At a second stage (validation of the panel and the lexicon), 5 of the previous samples were used to validate the sensory lexicon developed in this study and were randomly labeled as F8–F12. Three samples were used as taken from the wineries, while, to simulate common wide defects, another two of them (randomly selected) were spiked with concentrations of SO_2 (sensory descriptor sulfur) and ethyl acetate (sensory descriptor glue) above their detection thresholds. The concentrations used were 250 mg SO_2 L^{-1} (sample F10) (which maximum legal value is 200 mg·L^{-1} [7]) and 20 mg·L^{-1} of ethyl acetate (sample F12) (which detection threshold is 12.27 mg·L^{-1} [8]) and it is reported to range between 8.64 and 17.24 mg·L^{-1} in alcoholic beverages [9]. Finally, these two samples (F10 and F12) were left for 1 week in a hot room (reaching temperatures up to 35–40°C) to induce slight deterioration of the wines, by simulating real conditions of the wines in hot regions, such as Spain, with no proper control of the storage temperature.

2.2. Quality Parameters. The main physicochemical quality parameters (total alcohol content, volatile and total acidity, pH, relative density, total dry extract, total SO_2, and reducing sugars) were analyzed according to the official methods of the International Organisation of Vine and Wine (OIV), in accordance with the methods published in the first paragraph of Article 120g of Council Regulation (EC) No. 1234/2007 (published in accordance with Article 15 of Commission Regulation (EC) No. 606/2009 of 10 July 2009, and can be located at the OIV web page) [10].

2.3. Volatile Composition. The method selected to determine the composition and quantify the volatile profile of *Fondillón* samples was headspace solid phase microextraction (HS-SPME). For this analysis, 5 mL of wine, 1.5 g of NaCl, and 10 mL of ultrapure water were placed in a 50 mL vial with a polypropylene cap and a PTFE/silicone septum. The samples were equilibrated for 15 min at 40°C on the vials, and a DVB/CAR/PDMS fiber (50/30 μm) was exposed to the sample headspace at 40°C for 50 min. The extraction conditions (HS-SPME) were optimized to obtain a volatile profile positively correlated with sensory odor characteristics [11]. Similar extraction procedures have been successfully used in fruit liquors [12, 13] and pomegranate wine [14].

The isolation and identification of the volatile compounds were performed using the GC-MS conditions previously described [13]. A gas chromatograph, Shimadzu GC-2010, with a flame ionization detector (FID) was used for the quantification of the volatile composition of samples. The column and chromatographic conditions were the same as those reported previously by Gironés-Vilaplana et al. [13]. The extraction experiments and volatile studies were run in triplicate.

The proposed internal standard, benzyl acetate, was checked for its suitability for our GC analyses. It was found to be absent in the volatile profiles of *Fondillón* samples, it did not react with water, it possessed similar FID and MS response factors to most of the wine volatiles, and its chromatographic peak did not overlap with any of those of

the wine volatiles. Therefore, this compound (50 μL) was used as internal standard (concentration 1.0 g·L^{-1}).

Calibration curves were performed with the following compounds (Sigma-Aldrich, Madrid, Spain) as representative of each chemical family, and with intermediate molecular weights: octanoic acid (organic acids), 1-hexanol (alcohols), nonanal (aldehydes), ethyl hexanoate (esters), limonene (monoterpenes), and γ-nonalactone (lactones), and specific calibration curves were prepared for the two key compounds under study, TDN (1,1,6-trimethyl-1,2-dihydronaphthalene) and vitispirane. These calibration curves were done using synthetic wine as matrix; this wine was prepared by diluting 3.5 g of tartaric acid and 160 mL of ethanol with Milli-Q quality water until 1 L and, then, pH was adjusted to pH 3.5 [15, 16]. The correlation coefficients (R^2) for all compounds were above 0.995, and results were expressed as μg·L^{-1}.

2.4. Descriptive Sensory Analysis with Trained Panel. Fifteen panelists (5 women and 10 men) aged 24–61 years (mean age 38 years) participated in this study which took place at the facilities of the Regulatory Board of the Alicante Protected Designation of Origin, Alicante PDO, in Alicante (Spain). They were (i) selected (3 sessions of 1.5 h), according to their results in previous sensory discrimination, ranking, and recognition tests, (ii) trained (12 sessions of 1.5 h, during 4 months) (they were fully trained in descriptive sensory of wines from the Alicante PDO), and (iii) validated (2 sessions of 1.5 h), and are included in the control tools of the Regulatory Board to control the quality of their wines; this tool is included among those certified by the ISO/IEC 17065 : 2012 [17], with the reference number 118/C-PR198. These panelists are paid for their involvement in the current study and any other evaluations they perform.

No orientation session was needed because the panelists of the Alicante PDO are used to evaluate this type of wine. During the panel training, the panelists were instructed about the tasting protocol, the questionnaire structure and the order of the attributes to be evaluated, the lexicon (Table 1), and the scale to be used.

2.4.1. Wine Evaluation. Initially, wine samples of ~35 mL were served in the official "black" wine tasting cup [18] for the evaluation of the flavor of the samples, including positive and defect attributes. Later, ~20 mL was served in the official "transparent" wine-tasting cup [18]. It was decided that the visual stage of the evaluation should be conducted at the end of the tasting to avoid any influence on the objective description of the wines [19–21]. Samples were evaluated at room temperature (20 ± 1°C) and under white light and were served coded randomly with three digits together with the appropriate questionnaire, one at a time, and waiting 5 min between samples. Between samples and for palate cleansing, water and unsalted crackers were provided to panelists. In each questionnaire, panelists were asked, to evaluate the intensity of the following attributes: flavor (alcohol, fruity, floral, vegetable, spicy, animal, and toasted), basic tastes (sweet, sour, and bitter), chemical feelings (astringent), global attributes (imbalance and persistence), appearance (limpidity, color, and color intensity), and defects (vegetal, rotten apple, vinegar, glue, soap, sulfur, rotten egg, onion, cauliflower, horse, earthy, and cork). The intensity of the most relevant defect was scored, but the sensory descriptors of "all" found defects were marked in the tasting questionnaire. Panelists used an 11-point scale for the evaluation, in which 10 was extremely high intensity and 0 was extremely low or nonperceptible intensity. Reference materials for each attribute were prepared and were available for all panelists.

Evaluations were carried out in three 1 h sessions to have 3 replications. In each session, the 7 *Fondillón* samples under evaluation were monadically presented according to a William's Latin Square design balanced for order and carryover effects.

The panel was validated by analyzing five *Fondillón* samples, two of which were spiked with chemicals (SO_2 and ethyl acetate) leading to odor/aroma and flavor defects, as previously described. Besides, in each session, reproducibility (1 wine from a previous session is evaluated again) and repeatability (1 wine is evaluated twice in each session) are checked, and 1 wine with a significant defect is also introduced. These are the general rules for the working protocol of this accredited panel.

2.5. Affective Sensory Analysis with Consumers' Panel. A sample group of 60 consumers was recruited at Miguel Hernández University of Elche, UMH (Spain), and consisted of 25 men and 35 women aged between 22 and 67 years. Consumers lived in the East of Spain (Valencian Community, Murcia Region, Andalucia, and Castilla La Mancha Community). The main requirement for their recruitment was that they consumed alcoholic beverages, mainly "aged" wine, at least once a month. The consumer study was conducted at UMH during 4 sessions (15 consumers *per* session). In each session, consumers tested the 7 *Fondillón* samples; the 7 samples (F1–F7) under evaluation were monadically presented according to a William's Latin Square design balanced for order and carry-over effects. Twenty millilitre samples (along with the appropriate questionnaire) were served at room temperature (20 ± 1°C), coded with 3-digit numbers, one at a time, and with a 5 min gap between samples. Between samples and for palate cleansing, water and unsalted crackers were provided to panelists.

In each questionnaire, consumers were asked about their satisfaction degree for the *Fondillón* samples, using 9-point hedonic scale (9 = like extremely, 5 = neither like or dislike, and 1 = dislike extremely). Besides, consumers were also asked to rank samples according to their preference, from the least preferred sample to the most preferred one.

2.6. Statistical Analysis. All data included in this study are the mean of, at least, 3 replicates for the physicochemical parameters, 15 for descriptive sensory data, and 60 for affective data. All the data were first subjected to analysis of variance (ANOVA) and later to a multiple range test (Tukey's test), using StatGraphics Plus 5.0 software

Table 1: Main physicochemical parameters defining the quality of the *Fondillón* wine.

Parameters	ANOVA	F1	F2	F3	F4	F5	F6	F7
Total alcohol content (% v/v)	**	21.13 a‡	18.63 ab	20.17 a	16.97 b	16.60 b	16.16 b	18.50 ab
Volatile acidity (g acetic acid L^{-1})	*	0.71 b	0.62 b	1.05 b	1.03 b	0.75 b	0.99 b	1.50 a
Total acidity (g tartaric acid L^{-1})	*	5.30 b	5.46 b	5.50 b	7.80 a	6.50 a	7.27 a	6.60 a
pH	*	3.56 b	3.25 b	3.36 b	3.54 b	3.26 b	3.45 b	3.80 a
Relative density (20°C)	NS	1.0183	0.9986	1.0041	0.9935	1.0052	1.0077	0.9978
Total dry extract ($g{\cdot}L^{-1}$)	*	100 a	56.2 b	68.5 b	71.4 b	65.7 b	65.2 b	54.7 b
Total SO_2 ($mg{\cdot}L^{-1}$)	**	76.0 b	35.0 c	48.0 c	53.0 c	124 a	142 a	78.0 b
Reducing sugars ($g{\cdot}L^{-1}$)	**	39.70 a	29.30 b	39.20 a	23.00 c	34.50 ab	37.98 a	22.00 c

NS = not significant at $p < 0.05$; * and ** significant at $p < 0.05$, and 0.01, respectively. ‡Values (mean of 3 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.

(Manugistics, Inc., Rockville, MD). Differences were considered statistically significant at $p < 0.05$.

3. Results and Discussion

3.1. Quality Parameters. This is a type of wine with a high alcohol content; the minimum legal threshold is 16% (v/v) [7], and the experimental values ranged from 16.2 upto 21.2% (v/v) (Table 1).

The *Fondillón* wine is prepared with overripe Monastrell grapes. Then, the reducing sugars should be above a content of $15\,g{\cdot}L^{-1}$; although there is no legislation for this minimum value, there is an official maximum threshold for the content of sugars, $40\,g{\cdot}L^{-1}$ [7]. As can be seen in Table 1, the experimental values found for the total reducing sugars ranged from 22.0 up to $39.7\,g{\cdot}L^{-1}$ (mean of $32.24\,g{\cdot}L^{-1}$), proving that all samples used overripe grapes, with a very high content of initial sugars. It is interesting to mention that, in some cases, it is believed that there is an inverse relationship between the alcohol and the reducing sugar contents; however, this was not the case of *Fondillón* samples, and these two parameters showed no significant relationship ($R^2 = 0.1044$).

The total acidity of *Fondillón* should be above 3.5 g tartaric acid L^{-1} and had a mean of $6.35\,g{\cdot}L^{-1}$ (range between 5.30 and $7.80\,g{\cdot}L^{-1}$) in the studied samples. The legislation also establishes maximum values for the volatile acidity and total SO_2 at 1.50 g acetic acid L^{-1} and $200\,mg{\cdot}L^{-1}$, respectively, and the experimental ranges for these two parameters were $0.62–1.50\,g{\cdot}L^{-1}$ and $35–142\,mg{\cdot}L^{-1}$, respectively (Table 1).

As a summary of this section, it can be stated that the 7 wine samples analyzed fulfilled all legal requirements and, then, they can be legally classified and sold as *Fondillón* and, then, have Alicante PDO label.

3.2. Volatile Profile and Composition. Forty volatile compounds were isolated, identified, and quantified in the headspace of the seven *Fondillón* samples analyzed using HS-SPME (Table 2). The volatile aroma compounds found in this specific type of Spanish wine can be grouped in 8 chemical groups: (a) *esters* (17 compounds): e.g., ethyl acetate, ethyl propionate, and ethyl 2-methylpropanoate; (b) *alcohols* (7 compounds): e.g., isoamyl alcohol, 2,3-butanediol, and 1-hexanol; (c) *aldehydes* (5 compounds): e.g., benzaldehyde, nonanal, and decanal; (d) *terpenes* (4 compounds): e.g., α-thujene, α-pinene, and limonene; (e) *organic acids* (3 compounds): e.g., acetic acid, octanoic acid, and decanoic acid; (f) *ketones* (1 compound): β-methyl-γ-octalactone; (g) *sulfur compounds* (1 compound): sulfur dioxide; and (h) *others* (2 compounds): vitispirane and TDN (1,1,6-trimethyl-1,2-dihydronaphthalene). The mean relative abundance of these 8 chemical families in the *Fondillón* samples under study was

$$\begin{aligned}&\text{lactones}\,(0.13\%) < \text{terpenes}\,(0.85\%) < \text{others}\,(0.94\%)\\ &< \text{sulfur compounds}\,(1.41\%) < \text{organic acids}\,(2.24\%)\\ &< \text{aldehydes}\,(2.49\%) < \text{alcohols}\,(20.6\%) < \text{esters}\,(71.4\%)\end{aligned} \quad (1)$$

The overripe character of the Monastrell grapes used for the preparation of the *Fondillón* wine together with a long aging determined the volatile composition of the final wine, which was dominated by the ester family ($71.4 \pm 2.9\%$), followed by alcohols ($20.6 \pm 2.2\%$), as a result of the high alcoholic content of this type of wine. However, the most abundant group, esters, was not the key chemical group in determining the drivers for consumers' satisfaction degree, which was basically linked to the content of alcohols, according to a preliminary consumer study. There was a statistically significant negative correlation between the percentages of esters and alcohols ($R^2 = 0.9134$); that is, the higher the esters, the lower the alcohols.

The main volatile aroma compounds found in the *Fondillón* samples, their relative abundance, and their sensory descriptors were as follows:

(i) Diethyl succinate (mean for all 7 samples of 22.7%; descriptors: grape, fruity, wine)

(ii) Ethyl octanoate (15.0%; descriptors: apricot, floral) and ethyl acetate (17.7%; descriptors: anise, ethereal)

(iii) Isoamyl alcohol (9.8%; descriptors: whiskey) and phenethyl alcohol (7.2%; descriptors: honey, rose)

The most important esters in wines (e.g., ethyl acetate and ethyl octanoate) are considered to be the fatty acid ethyl esters, while branched-chain higher alcohols, including isoamyl alcohol, are synthesized from branched-chain amino acids [23]. Thus, there is nothing unusual in the main aroma compounds found in *Fondillón* wines. Similar to findings by Bailly et al. [24] in Sauternes wines, *Fondillón*

Table 2: Identification and contents ($\mu g \cdot L^{-1}$) of volatile compounds in *Fondillón* wine.

Volatile compounds	Code	Descriptor	RT (min)	RI		ANOVA	F1	F2	F3	F4	F5	F6	F7
				Exp.	Lit.[‡]		Concentration ($\mu g \cdot L^{-1}$)						
Sulfur dioxide	V1		4.48	na	na	**	139 b[Y]	16 d	14 d	163 b	292 a	61 c	38 cd
Acetic acid	V2	Vinegar	4.83	na	na	***	70 b	49 b	73 b	56 b	632 a	49 b	64 b
Ethyl acetate	V3	Anise, ethereal	5.14	na	na	***	737 c	746 c	959 c	631 c	3655 a	339 d	1733 b
Ethyl propanoate	V4	Pineapple, wine	5.58	712	714	***	9 d	19 d	28 d	20 d	119 c	353 b	1126 a
Isoamyl alcohol	V5	Whiskey	6.03	735	732	***	836 b	1262 a	1057 a	474 c	1100 a	106 d	27 d
Ethyl 2-methylpropanoate	V6		6.49	759	747	NS	20	18	23	12	172	8	125
(*Z*,*Z*)-2,3-Butanediol[¥]	V7		6.84	776	782	NS	21	23	24	50	38	9	88
(*E*,*E*)-2,3-Butanediol[¥]	V8		7.24	797	803	*	14 b	41 b	136 a	14 b	19 b	6 b	152 a
Ethyl lactate	V9	Butter, fruity	7.55	812	815	NS	184	155	123	231	97	35	230
Ethyl 2-methylbutyrate	V10	Apple, green, plum	8.19	845	847	NS	23	54	69	61	33	28	62
Ethyl 3-methylbutyrate	V11	Apple, green, plum	8.26	848	853	NS	31	54	51	53	51	19	56
1-Hexanol	V12	Green, herb	8.61	866	864	NS	33	54	40	74	38	29	48
Isoamyl acetate	V13	Banana, pear	8.76	874	875	*	76 b	72 b	66 b	103 a	89 ab	27 c	122 a
α-Thujene	V14		10.42	938	930	**	2 c	33 b	10 b	1 c	85 a	2 c	2 c
α-Pinene	V15	Woody	10.55	943	940	***	0 c	0 c	22 b	0 c	126 a	0 c	1 c
Benzaldehyde	V16	Almond, cherry	11.68	981	980	***	2 c	141 a	132 a	65 b	18 c	65 b	104 ab
Ethyl hexanoate	V17	Fruity, wine	12.32	1002	1000	NS	202	271	216	389	253	158	284
Limonene	V18	Citrus	13.78	1040	1033	NS	21	31	18	21	29	17	21
cis-β-Ocimene	V19		14.78	1066	1059	NS	5	8	5	5	8	5	4
Ethyl heptanoate	V20	Berry, fruity	16.16	1101	1100	NS	20	9	13	19	11	8	15
Ethyl sorbate	V21	Fruity, ethereal	16.40	1106	1111	***	557 a	14 b	20 b	2 b	2 b	4 b	42 b
Nonanal	V22	Fruity, nutty, citrus	16.77	1115	1112	NS	41	40	18	72	29	38	38
Phenethyl alcohol	V23	Honey, rose	17.48	1131	1127	*	416 c	589 b	423 c	617 a	706 a	270 c	550 bc
Octanoic acid	V24	Oily	19.48	1176	1180	NS	31	47	36	36	97	11	35
Benzyl acetate	Internal standard		19.53	1177	1168								
1-Nonanol	V25	Citrus, rose	19.66	1180	1173	NS	13	37	26	25	37	15	14
Diethyl succinate	V26	Grape, fruity, wine	19.98	1187	1191	**	1051 b	1842 a	1643 ab	1949 a	1970 a	729 b	2053 a
Ethyl octanoate	V27	Apricot, floral	20.78	1205	1204	***	637 b	709 b	757 b	1959 a	1701 a	727 b	968 b
Decanal	V28	Floral, citrus	21.42	1219	1212	NS	28	27	17	39	19	29	22
Ethyl-2-phenyl acetate	V29		23.34	1260	1255	NS	11	16	21	29	24	10	22
Phenethyl acetate	V30	Fruity, grape, wine	23.92	1273	1265	NS	14	14	11	23	31	4	20
Vitispirane	V31	Camphor, eucalyptus	25.34	1303	1286	***	26 c	20 cd	83 a	63 b	8 d	19 cd	46 b
Ethyl nonanoate	V32	Fruity, nutty	25.48	1306	1297	**	1 b	0 b	2 b	22 a	10 b	1 b	0 b
γ-Nonalactone	V33	Whiskey	27.23	1344	1344	*	1 b	3 b	6 b	10 b	44 a	6 b	7 b
Decanoic acid	V34	Fatty, citrus	28.60	1374	1373	NS	1	9	6	4	15	0	4
TDN	V35	Petroleum	29.06	1384	1367	***	1 d	1 d	53 a	39 b	16 c	12 c	48 ab
Ethyl decanoate	V36	Grape, oily	30.19	1409	1405	***	24 d	21 d	40 d	237 b	548 a	81 c	48 cd
Dodecanal	V37	Herb, floral	31.05	1428	1420	**	3 b	2 b	4 b	10 b	81 a	3 b	2 b
(*Z*)-4-dodecenol[¥]	V38		31.78	1445	1457	*	21 c	54 b	70 ab	76 ab	119 a	20 c	49 b
Ethyl dodecanoate[¥]	V39	Green, fruity, floral	39.12	1615	1598	NS	1	0	0	2	23	1	1
Tetradecanal[¥]	V40		40.13	1640	1625	NS	2	1	1	3	25	1	1
Total ($mg \cdot L^{-1}$)						***	5.33 c	6.50 b	6.32 b	7.66 b	12.4 a	3.30 c	8.27 b

[¥]Tentatively identified; RT: retention time; RI: retention indexes; Exp.: experimental; Lit.: literature. [‡]reference [22]. NS = not significant at $p < 0.05$; *, **, and *** significant at $p < 0.05$, 0.01, and 0.001, respectively. [Y]Values (mean of 3 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.

samples, after a minimum aging period of 10 years, still contained odorants found in young Monastrell wines, such as varietal aroma (α-pinene, limonene), fermentation alcohols (phenethyl alcohol), and esters (ethyl acetate, ethyl propanoate, ethyl 2-methylpropanoate), but also contained maturation-related compounds (γ-nonalactone, vitispirane, TDN). Vitispirane and TDN are norisoprenoids that could come from the degradation of carotenoid molecules during wine aging [25].

Factors such as oxygen, temperature, and pH are key parameters, influencing the oxidative changes of *Fondillón* during its prolonged aging in oak vats, which are permeable to the entrance of oxygen. The specific volatile compounds that develop during its aging are what control the commercial value of the *Fondillón* wines. Five compounds were key for the aroma quality of Port wine, and their concentrations were markedly different between young and aged samples [26]. These compounds were β-damascenone (sensory descriptor: rose and citrus), β-ionone (floral, violet and rose), 2,2,6-trimethylcyclohexanone, TCH (rose), 1,1,6-trimethyl-1,2-dihydronaphthalene, TDN (petroleum), and vitispirane (camphor and eucalyptus). Some of these norisoprenoid molecules were responsible for floral and violet notes at low concentrations; however, some others (TDN and vitispirane) have nonpleasant aroma notes (e.g., petroleum or camphor), especially at high concentrations, but have been correlated positively correlated with the age of Port wine [27]. There were 15, 5, and 3 times higher levels of TDN, vitispirane, and TCH in 40-year-old than in young ports [26]. In the *Fondillón* samples, and under the working conditions assayed (HS-SPME and DVB/CAR/PDMS fiber), only 2 of these compounds, vitispirane and TDN, were found using HS-SPME. Future studies will be conducted using other extraction techniques and SPME fibers to check whether all these 5 compounds can also be found in *Fondillón*. The levels of these two compounds (vitispirane and TDN) were positively correlated ($R^2 = 0.8410$ and 0.7797, respectively) with the age of the solera and can be initially considered a good indicator of the age of the *Fondillón* samples. Besides, there is a need for further research to determine the key odorants in this special Alicante wine, by using gas chromatography and olfactometry [28, 29].

3.3. Descriptive Sensory Analysis with Trained Panel. Legal sensory definition of the *Fondillón* wine [7] is as follows:

(i) *Color*: mahogany and amber and with copper tones

(ii) *Nose*: aromatically intense, ripe fruit nuts, well-integrated wood, high roasted

(iii) *Taste*: balanced, good structure, big volume, persistent, and slight sweet

This definition is certainly not wide enough to fully express the whole personality of this type of wine. Besides, there is a pressing need to have methods certified by official accreditation bodies to score the sensory quality of foods [30], and wine is not an exemption. In 2015, the Alicante PDO selected, trained, and validated their sensory panel to evaluate the wines protected by this organization. During the training, the panel developed, together with the UMH researchers who were responsible for this training, the lexicon compiled in Table 3. This lexicon was prepared according to experience of the panelists included in the panel, who were oenologists, sommeliers, researchers, etc., and to previous studies developing similar lexicons for other Spanish wines, such as Rioja Alavesa [19, 31] and txakoli [32]. The lexicon was divided into 4 phases or steps: (i) flavor (including odor (perception of volatile compounds with the wine in the cup) and aroma (perception of the volatile compounds with the wine in the mouth)), (ii) global, (iii) visual, and (iv) defects for each one of the previous three phases. The visual evaluation was conducted in a black cup to avoid any subjective color bias.

The Alicante PDO has prepared "typical profiles" for each one of the wines under their protection, including *Fondillón*, and the wines under evaluation should be as close to the Alicante profiles as possible, with a tolerance level established by the Regulatory Body. The sensory profile of the first 7 *Fondillón* samples (F1–F7) fully agreed with the scores of the typical *Fondillón* profile of the Alicante PDO, shown in Table 4, in the column "PDO profile."

However, the trained panel of the Alicante PDO, using the sensory lexicon specific for *Fondillón* (Table 3), identified two samples (F10 and F12) during the validation step, which were considered as having significant problems, which should preclude their labeling with the Alicante PDO seal (Table 4). The problems in these two samples were mainly due to (i) defects in the olfactory phase, with defects having scores of 2.8 and 2.5 and (ii) imbalances (sour, astringent, and bitter) in the global phase, with scores being 3.0 and 2.0, for samples F10 and F12, respectively. Eleven out of 15 panelists described the defect found in the sample F10 (SO_2) as sulfur, while all panelists properly described the excessive occurrence of ethyl acetate as "glue."

Besides, two parameters were used to validate the quality of the sensory lexicon and the performance of the sensory panel: (i) repeatability in attribute identification and scores: ability to identify the same attributes (including defects) and give similar scores when the same wine is evaluated in two replications in the same session and (ii) reproducibility in attribute identification and scores: ability to identify the same attributes (including defects) and give similar scores when the same wine is evaluated in replicate in different sessions [19]. The values of these two parameters for the panel sessions were acceptable for the requirements of the PDO Alicante; standard deviation of the same wine sample should be ≤ 1.3 for all the identified attributes.

Thus, the conclusion of this section was that the sensory lexicon and questionnaire developed especially for *Fondillón* samples under the Alicante PDO have been validated by detecting the two spiked and spoiled samples.

3.4. Affective Sensory Analysis with Consumers' Panel. A preliminary consumer study (with only 60 consumers) seemed to indicate that the highest satisfaction degree (8.0 in

Table 3: Lexicon used for the descriptive analysis of *Fondillón*.

Attributes	Definition	References and intensities
Flavor		
Alcohol	A flavor reminiscent of alcoholic compounds	Ethanol solution 7% = 2.0; ethanol solution 11% = 5.0; ethanol solution 18% = 9.5
Fruity	A flavor blend that is sweet and reminiscent of a variety of fruits	Citral 16 $\mu g \cdot L^{-1} = 6.0$; isoamil acetate 30 $\mu g \cdot L^{-1} = 6.0$; benzaldehyde 100 $\mu g \cdot L^{-1} = 6.0$
Floral	A sweet, heavy aromatic blend of a combination of flowers	Geraniol 10 $\mu g \cdot L^{-1} = 6.0$; β-ionona 0.10 $\mu g \cdot L^{-1} = 6.0$
Vegetable	Flavor reminiscent of a variety of different vegetables	2-Isobutyl-3-methoxypyrazine 0.02 $\mu g \cdot L^{-1} = 6.0$; *cis*-3-hexen-1-ol 70 $\mu g \cdot L^{-1} = 6.0$; 1-octen-3-ol 1 $\mu g \cdot L^{-1} = 6.0$
Spicy	Flavor reminiscent of different species, which are directly related to the passage of wine barrels	Eugenol 15 $\mu g \cdot L^{-1} = 6.0$; anethole 70 $\mu g \cdot L^{-1} = 6.0$
Animal	Flavor reminiscent of animals or products derivatives thereof	Albona butter flavor 6 $\mu g \cdot L^{-1} = 6.0$; "le nez du vin" flavor no. 45 = 9.0
Toasted	Aromas reminiscent of roasted products and generally coming from the toasting of the barrels	Vainillin 20 $\mu g \cdot L^{-1} = 6.0$; 2-acetylthiazole 5 $\mu g \cdot L^{-1} = 6.0$
Sweet	The fundamental taste factor associated with a sucrose solution	Sucrose solution 4% = 2.5; sucrose solution 8% = 5.0; sucrose solution 16% = 9.5
Sour	The taste stimulated by acids, such as citric and malic.	Tartaric acid solution 0.05% = 2.5; tartaric acid solution 0.08% = 4.0; tartaric acid solution 0.20% = 9.5
Bitter	The taste stimulated by substances such as quinine or caffeine	Caffeine solution 0.05% = 2.5; caffeine solution 0.08% = 4.0; caffeine solution 0.20% = 9.5
Astringent	The complex of drying, puckering, and shrinking sensations in the oral cavity	Alum solution 0.05% = 1.5; alum solution 0.10% = 3.0; alum solution 0.20% = 6.0
Global		
Imbalance	Wine attribute or attributes that prevail over the rest, breaking the balance	Sour: tartaric acid 2 $g \cdot L^{-1} = 6$; astringent: tannin 4 $g \cdot L^{-1} = 6$; bitter: quinine sulphate 0.03 $g \cdot L^{-1} = 6$; alcohol: ethanol 60 $mL \cdot L^{-1} = 6$
Persistence	Time it remains in the mouth, the characteristic flavor of the fruit after swallowing the sample	5–8 s = 5.0; 15–18 s = 10
Visual		
Limpidity	Without particles or coloidal elements in suspension	Isolated elements = 5; without particles = 10
Color	Visual evaluation of the color intensity of the sample	Pantone 1675C = 2.0; pantone 201C = 4.0; pantone 200C = 6.0
Color int.	Depth of color when you put a text under the glass	If you can read the text = 1.0; if you can see the text but you can't read it = 5.0; if you can't see the text = 10
Defects		
Vegetal	Defect caused by immature grapes or insufficient cleaning of bunches	"Le nez du vin, faults" no. 1 = 8
Rotten apple	Wine oxidation by *Candida mycoderma*, with formation of acetaldehyde	"Le nez du vin, faults" no. 2 = 8
Vinegar	Formation of acetic acid by *Gluconobacter* and *Acetobacter*	"Le nez du vin, faults" no. 3 = 8
Glue	Formation of ethyl acetate by reaction of acetic acid with ethanol	"Le nez du vin, faults" no. 4 = 8
Soap	Soapy notes caused by the salts of certain fatty acids, mainly decanoic acid	"Le nez du vin, faults" no. 5 = 8
Sulfur	Sulfurous notes from too much sulfite.	"Le nez du vin, faults" no. 6 = 8
Rotten egg	Formation of hydrogen sulfide by reduction of sulfiting by yeasts.	"Le nez du vin, faults" no. 7 = 8
Onion	Ethanethiol formation by reaction of H_2S with ethanol	"Le nez du vin, faults" no. 8 = 8
Cauliflower	Note characteristic aromatic wines made from poorly debourbaged musts	"Le nez du vin, faults" no. 9 = 8
Horse	Unpleasant animal note (mainly phenolic) that resembles the horse stable smells. This defect may occur due to presence of the *Brettanomyces*	"Le nez du vin, faults" no. 10 = 8
Earthy	Notes that smells like wet earth	"Le nez du vin, faults" no. 11 = 8
Cork	Aromatic note caused by the poor quality of cork employed. This complex defect includes simple notes like solvents and moisture	"Le nez du vin, faults" no. 12 = 8

Table 4: Descriptive sensory analysis of commercial samples of *Fondillón* used to validate the sensory lexicon.

Attribute	ANOVA[†]	PDO profile	F8	F9	F10	F11	F12
			Sensory intensity (scale 0–10)				
Odor (o)							
Alcohol	NS	7.0	7.0	7.0	7.0	6.5	7.0
Fruity	*	6.0	6.0 a[‡]	6.0 a	5.5 b	6.0 a	4.0 c
Floral	NS	2.0	2.0	2.0	3.0	2.0	2.0
Vegetal	***	2.5	3.0 b	3.5 ab	4.0 a	2.0 c	2.5 b
Spicy	NS	3.5	2.5	4.0	4.0	3.0	3.5
Animal	**	3.0	3.0 b	3.0 b	4.0 a	2.8 b	3.0 b
Toasted	NS	6.0	5.5	7.0	5.0	6.3	6.0
Defects	***	0	0 b	0 b	2.8 a	0 b	2.5 a
Flavor (f)							
Alcohol	NS	7.0	7.0	7.0	7.0	7.0	7.0
Fruity	***	6.0	6.0 a[‡]	6.5 a	4.0 b	6.0 a	3.0 b
Floral	NS	2.0	2.0	1.0	2.0	2.0	2.0
Vegetal	NS	2.0	2.0	2.0	2.0	2.0	3.0
Spicy	NS	4.0	3.0	4.0	4.0	2.8	4.0
Animal	NS	3.0	3.0	2.0	3.0	2.0	3.0
Toasted	NS	6.0	6.0	6.8	5.0	6.3	6.0
Sweet	***	3.0	2.0 b	5.0 a	3.0 b	4.0 ab	3.0 b
Sour	**	4.0	4.0 ab	3.0 b	5.0 a	4.0 ab	5.0 a
Bitter	NS	2.0	2.0	2.0	2.0	1.3	3.0
Astringent	NS	2.0	2.0	2.0	3.0	1.0	2.5
Defects	**	0	0 c	0 c	2.5 a	0 c	1.0 b
Global							
Imbalances	***	0	1.0 b	0 c	3.0 a	0 c	2.0 ab
Aftertaste	*	7.0	7.0 ab	8.0 a	6.5 b	7.0 ab	6.0 b
Appearance (a)							
Limpidity	NS	9.0	9.0	9.0	8.5	9.0	8.0
Color (hue)	**	5.0	5.0 b	5.0 b	6.0 a	5.0 b	3.0 c
Color intensity	**	3.0	3.5 b	3.0 b	5.0 a	4.8 a	3.0 b
Defects	NS	0	0	0	0	0	0.5
Qualification			OK	OK	NOT OK	OK	NOT OK
Liking[¶]	***		6.0 b	8.0 a		6.7 b	
Ranking[¶]	***		b	a		b	

[†]NS = not significant at $p < 0.05$; *, **, and *** significant at $p < 0.05$, 0.01, and 0.001, respectively. [‡]Values (mean of 15 trained panelists) followed by the same letter, within the same row, were not significantly different ($p < 0.05$), according to Tukey's least significant difference test. [¶]Mean satisfaction degree of 30 consumers is denoted by liking, and statistical results of Friedman's test are denoted by ranking.

sample F2) was linked to the fruity notes, the alcoholic content, the aftertaste, and the presence of key volatile compounds, such as vitispirane (which sensory descriptor is eucalyptus) and benzaldehyde, with a bitter almond note. The satisfaction degree ranged between 4.4 and 8.0. However, more complex affective tests (regular *Fondillón* consumers, a consumer number >100, and 4-5 locations in different regions of Spain) must be conducted to prove the hypothesis raised in this preliminary affective study.

4. Conclusions

The combined use of instrumental (HS-SPME-GC-MS/FID) and sensory (descriptive sensory analysis and consumer studies) tools has allowed proper classification of the *Fondillón* samples. To have a full description of this wine, a specific lexicon to describe wines under the Alicante PDO label was developed. This wine (*Fondillón*), historically known as Alicante wine, was highly appreciated by today's Spanish consumers when having intense fruity notes, but at the same time, high alcoholic content and some bitter and balsamic notes, such as those coming from benzaldehyde (bitter almond) and vitispirane (eucalyptus). However, further affective studies are needed using a higher number of consumers and including more locations within different regions of Spain and also in the European Union as the initial potential market for this wine. The age of the *Fondillón* samples has been successfully linked with the contents of two key compounds TDN and vitispirane, but other extraction and analysis techniques must be assayed to fully prove this statement.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

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References

[1] P. Reboredo-Rodríguez, C. González-Barreiro, R. Rial-Otero, B. Cancho-Grande, and J. Simal-Gándara, "Effects of sugar concentration processes in grapes and wine aging on aroma compounds of sweet wines-a review," *Critical Reviews in Food Science and Nutrition*, vol. 55, no. 8, pp. 1053–1073, 2015.

[2] L. Rolle, F. Torchio, S. Giacosa, and V. Gerbi, "Modifications of mechanical characteristics and phenolic composition in berry skins and seeds of mondeuse winegrapes throughout the on-vine drying process," *Journal of the Science of Food and Agriculture*, vol. 89, no. 11, pp. 1973–1980, 2009.

[3] A. Valero, S. Marín, A. J. Ramos, and V. Sanchis, "Survey: ochratoxin a in european special wines," *Food Chemistry*, vol. 108, no. 2, pp. 593–599, 2008.

[4] M. Figuereido-González, B. Cancho-Grande, and J. Simal-Gándara, "Effects of colour and phenolic composition of sugar concentration processes in dried-on-or dried-off-vine grapes and their aged or not natural sweet wines," *Trends in Food Science and Technology*, vol. 31, no. 1, pp. 36–54, 2013.

[5] EC, *Council Regulation (EC) No 479/2008 of April 2008 On the Common Organisation of the Market in Wine, in 479/2008*, European Union, Brussels, Belgium, 2008.

[6] D. Alicante, Fondillón de Alicante, time is a luxury, 2016, http://vinosalicantedop.org/fondillon/en/.

[7] G. Valenciana, *Orden 5/2011, de 16 de Noviembre, de la la Conselleria de Agricultura, Pesca, Alimentación y Agua, Por la que se Aprueba el Texto del Reglamento y Pliego de Condiciones de la Denominación de Origen Protegida Alicante y su Consejo Regulador*, Diario Oficial de la Comunitat Valenciana, Valencia, Spain, 2011.

[8] C. Lorenzo, F. Pardo, A. Zalacain, G. L. Alonso, and M. R. Salinas, "Differentiation of co-winemaking wines by their aroma composition," *European Food Research and Technology*, vol. 227, no. 3, pp. 777–787, 2008.

[9] G. A. Burdock, *Fenaroli's Handbook of Flavor Ingredients*, CRC Press, Taylor & Francis Group, Boca Raton, FL, USA, 6th edition, 2010.

[10] OIV, *Compendium of International Methods of Analysis of Wines and Musts*, vol. 2, OIV, Paris, France, 2016, http://www.oiv.int/en/technical-standards-and-documents/methods-of-analysis/compendium-of-international-methods-of-analysis-of-wines-and-musts-2-vol.

[11] A. Alonso, L. Vázquez-Araújo, S. García-Martínez, J. J. Ruiz, and Á. A. Carbonell-Barrachina, "Volatile compounds of traditional and virus-resistant breeding lines of Muchamiel tomatoes," *European Food Research and Technology*, vol. 230, no. 2, pp. 315–323, 2009.

[12] Á. A. Carbonell-Barrachina, P. J. Szychowski, M. V. Vásquez, F. Hernández, and A. Wojdyło, "Technological aspects as the main impact on quality of quince liquors," *Food Chemistry*, vol. 167, pp. 387–395, 2015.

[13] A. Gironés-Vilaplana, Á. Calín-Sánchez, D. A. Moreno, Á. A. Carbonell-Barrachina, and C. García-Viguera, "Novel maqui liquor using traditional pacharán processing," *Food Chemistry*, vol. 173, pp. 1228–1235, 2015.

[14] A. J. Andreu-Sevilla, P. Mena, N. Martí, C. García Viguera, and Á. A. Carbonell-Barrachina, "Volatile composition and descriptive sensory analysis of pomegranate juice and wine," *Food Research International*, vol. 54, no. 1, pp. 246–254, 2013.

[15] L. Vera, M. Mestres, R. Boqué, O. Busto, and J. Guasch, "Use of synthetic wine for models transfer in wine analysis by HS-MS e-nose," *Sensors and Actuators B: Chemical*, vol. 143, no. 2, pp. 689–695, 2010.

[16] A. de-la-Fuente-Blanco, M.-P. Sáenz-Navajas, and V. Ferreira, "Levels of higher alcohols inducing aroma changes and modulating experts' preferences in wine model solutions," *Australian Journal of Grape and Wine Research*, vol. 23, no. 2, pp. 162–169, 2017.

[17] ISO 17065:2012, *Conformity Assessment. Requirements for Bodies Certifying Products, Processes and Services in 17065: 2012*, ISO/IEC, New York, NY, USA, 2012.

[18] ISO 3591:1977, *Sensory Analysis. Apparatus. Wine Tasting Glass*, ISO, Geneva, Switzerland, 1977.

[19] I. Etaio, M. Albisu, M. Ojeda, P. F. Gil, J. Salmerón, and F. J. Pérez Elortondo, "Sensory quality control for food certification: a case study on wine. Panel training and qualification, method validation and monitoring," *Food Control*, vol. 21, no. 4, pp. 542–548, 2010.

[20] G. Morrot, F. Brochet, and D. Dubourdieu, "The color of odors," *Brain and Language*, vol. 79, no. 2, pp. 309–320, 2001.

[21] W. V. Parr, K. G. White, and D. A. Heatherbell, "The nose knows: influence of colour on perception of wine aroma," *Journal of Wine Research*, vol. 14, no. 2-3, pp. 79–101, 2003.

[22] NIST. National Institute of Standards and Technology, 2016, http://webbook.nist.gov/chemistry/name-ser.html.

[23] A. L. Robinson, P. K. Boss, P. S. Solomon, R. D. Trengove, H. Heymann, and S. E. Ebeler, "Origins of grape and wine aroma. Part 1. Chemical components and viticultural impacts," *American Journal of Enology and Viticulture*, vol. 65, no. 1, pp. 1–24, 2014.

[24] S. Bailly, V. Jerkovic, A. Meurée, A. Timmermans, and S. Collin, "Fate of key odorants in sauternes wines through aging," *Journal of Agricultural and Food Chemistry*, vol. 57, no. 18, pp. 8557–8563, 2009.

[25] A. Silvaferreira and P. Guedes de Pinho, "Nor-isoprenoids profile during port wine ageing—influence of some technological parameters," *Analytica Chimica Acta*, vol. 513, no. 1, pp. 169–176, 2004.

[26] N. Moreira and P. Guedes de Pintho, "Port wine," in *Advances in Food and Nutrition Research*, R. S. Jackson, Ed., pp. 119–146, Elsevier, Amsterdam, Netherlands, 2011.

[27] C. F. Ross, A. C. Zwink, L. Castro, and R. Harrison, "Odour detection threshold and consumer rejection of 1,1,6-trimethyl-1,2-dihydronaphthalene in 1-year-old riesling wines," *Australian Journal of Grape and Wine Research*, vol. 20, no. 3, pp. 335–339, 2014.

[28] R. M. Callejón, M. L. Morales, A. M. Troncoso, and A. C. Silva Ferreira, "Targeting key aromatic substances on the typical aroma of sherry vinegar," *Journal of Agricultural and Food Chemistry*, vol. 56, no. 15, pp. 6631–6639, 2008.

[29] L. Culleré, A. Escudero, J. Cacho, and V. Ferreira, "Gas chromatography–olfactometry and chemical quantitative study of the aroma of six premium quality Spanish aged red wines," *Journal of Agricultural and Food Chemistry*, vol. 52, no. 6, pp. 1653–1660, 2004.

[30] F. J. P. Elortondo, M. Ojeda, M. Albisu, J. Salmerón, I. Etayo, and M. Molina, "Food quality certification: an approach for the development of accredited sensory evaluation methods," *Food Quality and Preference*, vol. 18, no. 2, pp. 425–439, 2007.

[31] I. Etaio, M. Albisu, M. Ojeda, P. F. Gil, J. Salmerón, and F. J. P. Elortondo, "Sensory quality control for food certification: a case study on wine. Method development," *Food Control*, vol. 21, no. 4, pp. 533–541, 2010.

[32] I. Etaio, P. F. Gil, M. Ojeda, M. Albisu, J. Salmerón, and F. J. Pérez Elortondo, "Improvement of sensory quality control in PDO products: an example with txakoli white wine from Bizkaia," *Food Quality and Preference*, vol. 23, no. 2, pp. 138–147, 2012.