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Highlights

- Sparkling wines from Albarín, Verdejo, Godello and Prieto Picudo were the richest in volatiles.
- These four grape varieties were the most interesting to elaborate sparkling wines.
- Verdejo and Prieto Picudo sparkling wines presented the best foam characteristics.
- Sparkling wines maintain their varietal characteristics even after long aging time.

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Grape variety, aging on lees and aging in bottle after disgorging influence on volatile composition and foamability of sparkling wines

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ABSTRACT

The aim of this work was focused on the study of the influence of grape variety and aging time in contact with lees and without lees, on volatile composition and foamability of white and rosé sparkling wines. Seven different grape varieties were used and the sparkling wines were studied until 30 months of aging on lees and after 12 months of aging in bottle after disgorging.

Sparkling wines from *Albarín*, *Verdejo*, *Godello* and *Prieto Picudo* grape varieties were the richest in most of the volatile compounds analyzed, especially those that contribute to the fruity aroma of wines, and maintained their varietal characteristics even after long aging time (30 months). *Verdejo* and *Prieto Picudo* sparkling wines presented the best foam characteristics, followed by *Albarín* and *Godello* wines.

Considering all the results, *Albarín, Verdejo, Godello* and *Prieto Picudo* were the most interesting grape varieties to elaborate sparkling wines, following the traditional or "champenoise" method. © 2014 Published by Elsevier Ltd.

1. Introduction

Sparkling wines are obtained after a second fermentation of a base wine that can be carried out in closed bottles or in hermetically sealed tanks. High quality sparkling wines, such as Champagne wines in France, Cava wines in Spain or Talento in Italy, are fermented in closed bottles following the traditional or "champenoise" method, and they remain in contact with the yeast lees in a bottle for at least 9 months (EC Regulation N° 606/2009). The greatest differences among sparkling wines are mainly due to the grape varieties and the aging time on lees (Andres-Lacueva, Gallart, Lopez-Tamames, & Lamuela-Ravento;s, 1996; Pozo-Bayón, Martínez-Rodríguez, Pueyo, & Moreno-Arribas, 2009; Riu-Aumatell, Bosch-Fusté, López-Tamames, & Buxaderas, 2006; Torrens, Riu-Aumatell, Vichi, López-Tamames, & Buxaderas, 2010).

During the sparkling wine aging, yeast autolysis leads to significant changes in wine composition (Alexandre and Guilloux-Benatier, 2006), and especially in the volatile compounds that

E-mail address: permagsi@itacyl.es (S. Pérez-Magariño).

http://dx.doi.org/10.1016/j.lwt.2014.11.011 0023-6438/© 2014 Published by Elsevier Ltd. could have a great effect on the final quality of these wines (Francioli, Torrens, Riu-Aumatell, López-Tamames, & Buxaderas, 2003: Pozo-Bavon, Puevo, Martin-Alvarez, Martinez-Rodriguez, & Polo, 2003: Pozo-Bavón, Martín-Álvarez, Moreno-Arribas, Andujar-Ortiz, & Pueyo, 2010). During this process, different enzymatic and chemical reactions can lead to the formation or degradation of some volatile compounds, and others can be released into the wine (Del Barrio-Galán, Ortega-Heras, Sánchez-Iglesias, & Pérez-Magariño, 2012; Riu-Aumatell et al., 2006; Torrens et al., 2010), modifying the aroma profile of sparkling wines. On the other hand, some volatile compounds can be adsorbed on the yeast lees, reducing their concentration in sparkling aged wines, mainly the most hydrophobic ones (Gallardo-Chacón, Vichi, López-Tamames, & Buxaderas, 2009, 2010). Gallardo-Chacón et al. (2009) determined the volatile compounds retained by lees during the second fermentation of sparkling wines and found that esters, aldehydes and terpenes were retained by the lees surface. Sorption depends not only on the physicochemical characteristics of the volatile compounds but also on the structure of the yeast cell walls, hence the retention of volatile compounds by the lees surface can be reversible and the volatile composition of these wines can change over long aging time (Gallardo-Chacón et al., 2010). Therefore, the final aging time will determine the type and amount of the volatile compounds present in sparkling wines (Francioli et al., 2003;

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Table 1

Volatile compounds of the sparkling wines at the different aging times: T9, T18, T30: nine, eighteen, thirty months of aging on lees; 12 MB: twelve months in bottle after disgorging. Data in mg/L except those marked with an asterisk that are expressed in µg/L.

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bioama constant Lindor Lindor	Ethyl decanoate	0.068	0.052	0.036	0.084	0.027	0.059	0.058	0.040	0.077	0.035
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cis-3-heap-1-ol 0.163 0.163 0.173 0.130 0.130 0.136 0.006 0.002 0.006 Linalol* 5.418 7.73 0.008 1.854 0.063 2.748 1.664 0.014 0.140 0.052 Circonello* 9.446 1.103 0.577 0.812 0.493 1.577 1.419 0.447 0.821 0.577 -fubroidlactome* 1.358 9.353 8.033 1.033 1.157 1.419 0.447 0.821 0.577 1.415 1.107 2.438 2.082 -fubroidlactome* 2.755 2.111 2.502 2.626 1.221 1.07 1.07 1.837 1.614 Mathibur 1.65 1.04 1.255 1.64 1.17 7.3 3.93 1.14 7.40 Carbonalidine* 1.55 1.04 1.255 2.61 3.44 1.7 2.68 2.31 Carbonalidine* 1.55 1.04 1.25 1.618 1.17	trans-3-hexen-1-ol	0.115	0.202	0.141	0.091	0.124	0.080	0.132	0.085	0.052	0.078
	cis-3-hexen-1-ol	0.163	0.153	0.128	0.104	0.115	0.130	0.148	0.106	0.082	0.099
	Benzyl alcohol	0.106	0.114	0.104	0.120	0.102	0.119	0.136	0.103	0.102	0.106
$\begin{split} \hline \begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	Linalool*	5 418	1 739	0.808	1 854	0.693	2 746	1 664	0.614	1 414	0 582
$\begin{split} \begin{array}{c} \mbox{remerial} & 1.446 & 1.103 & 0.577 & 0.812 & 0.457 & 1.277 & 1.41 & 0.647 & 0.211 & 0.539 \\ -5.879 & 1.257 & 1.250 & 1.251 & 1.250 & 3.259 & 2.626 & 2.021 & 1.77 & 1.907 & 2.438 & 2.082 \\ -5.890 & 1.06 & 1.03 & 12.68 & 1.02 & 1.142 & 5.61 & 5.33 & 1.174 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 7.3 & 9.9 & 1.44 & 1.17 & 7.40 & 1.141 & 1.17 & 1.141 & 1.17 & 1.141 & 1.17 & 1.141 & 1.141 & 1.17 & 1.141 & 1.141 & 1.17 & 1.141 & 1.17 & 1.141 & 1.141 & 1.17 & 1.141 & 1.141 & 1.17 & 1.141 & 1.141 & 1.17 & 1.141 & 1.141 & 1.17 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.141 & 1.1$	a-Ternineol*	9 878	0 303	8 336	10.835	8 133	3 960	4 678	3 108	4 501	3 396
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Citronellol*	1 1/16	1 102	0.550	0.810	0.493	1 577	1 /10	0.647	0.821	0.500
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	v-Buturolactone	1.440	12 50	0.377	0.012	0.455	14.25	1.419	10.047	11 51	11 02
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	I-Dutyroidcione*	2 705	13.30 2 111	5.45 3.503	2.27	3.12	2 0 2 1	12.29	10.27	11.34	11.05
$ \begin{array}{c} \begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	y-inolididciolle	2.705	2.111 nd	2.302	5.299	2.020	2.021	1.///	1.907	2.438	2.082
	Vallilli	2./99	iid	11.00	9.260	17.24	1.949	nd	3.159	18.//	10.14
tetry valuate: nd	wethyl vanillate*	14.45	8.10	11.03	12.65	11.62	11.12	5.61	5.33	11./4	/.40
$ \begin{array}{c} \mbox{Acctivations} & 15.5 & 10.4 & 12.8 & 11.0 & 14.1 & 11.7 & 7.3 & 9.9 & 14.4 & 11.7 \\ 1-propanol & 28.3 & 23.8 & 21.0 & 25.2 & 26.1 & 34.4 & 27.6 & 25.9 & 34.5 & 31.1 \\ \text{isobutanol} & 22.3 & 19.7 & 19.0 & 19.8 & 19.5 & 24.0 & 24.0 & 21.7 & 26.8 & 23.4 \\ \text{isobutanol} & 17.9 & 17.5 & 18.2 & 166 & 187 & 188 & 166 & 19.3 & 20.2 & 196 \\ \mbox{-1} & 15.5 & 0.120 & 0.178 & 0.197 & 0.433 & 0.166 & 0.125 & 0.219 & 0.121 \\ \hline \\ $	Ethyl vanillate*	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Acetovanillone*	15.5	10.4	12.8	11.0	14.1	11.7	7.3	9.9	14.4	11.7
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	2-Phenylethanol	37.7	66.6	49.7	43.9	45.3	32.3	58.2	37.3	29.8	35.5
Isobutanol 22.3 19.7 19.0 19.8 19.5 24.0 21.7 26.8 23.4 4-vinylguaiacol 0.155 0.120 0.178 0.213 0.197 0.433 0.166 0.125 0.219 0.121 4-vinylguaiacol 0.155 0.120 0.178 0.213 0.197 0.433 0.166 0.125 0.219 0.121 Ethyl barytate 0.143 0.159 0.180 0.127 0.200 0.160 0.154 0.154 0.0178 0.0161 Ethyl 2-methylburytate 0.046 0.066 0.080 0.061 0.058 0.033 0.035 0.037 0.034 0.075 0.0164 0.075 0.017 0.017 0.0164 0.056 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085 0.085	1-propanol	28.3	23.8	21.0	25.2	26.1	34.4	27.6	25.9	34.5	31.1
	Isobutanol	22.3	19.7	19.0	19.8	19.5	24.0	24.0	21.7	26.8	23.4
	Isoamyl alcohols	179	175	182	166	187	188	186	193	202	196
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	4-vinylguaiacol	0.155	0.120	0.178	0.213	0.197	0.433	0.166	0.125	0.219	0.121
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		Godello					Viura				
		T9	T18	T30	T9+12 MB	T18 + 12 MB	T9	T18	T30	T9+12 MB	T18 + 12 MB
	Ethyl butyrate	0.143	0.159	0.180	0.127	0.200	0.160	0.154	0.154	0.178	0.161
Ethyl isovalerate 0.066 0.085 0.085 0.085 0.085 0.085 0.035 0.0379 0.374 Ethyl hexanoate 0.536 0.492 0.462 0.487 0.431 0.358 0.389 0.353 0.379 0.374 Ethyl locatnoate 0.566 0.520 0.468 0.511 0.408 0.312 0.353 0.302 0.359 0.310 Ethyl locatnoate 0.080 0.066 0.051 0.408 0.048 0.013 0.070 0.031 0.035 0.030 0.095 0.093 0.099 0.109 2-Phenylethyl acetate 0.048 0.018 0.017 0.043 0.027 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.026 0.016 0.014 0.017 0.075 0.118 <td< td=""><td>Ethyl 2-methylbutyrate</td><td>0.046</td><td>0.073</td><td>0.084</td><td>0.063</td><td>0.081</td><td>0.031</td><td>0.048</td><td>0.063</td><td>0.049</td><td>0.076</td></td<>	Ethyl 2-methylbutyrate	0.046	0.073	0.084	0.063	0.081	0.031	0.048	0.063	0.049	0.076
Ethyl hexanoate0.5360.4920.4620.4870.4310.3580.3890.3330.3790.374Ethyl lactate16.921.119.214.317.518.425.823.616.630.0Ethyl otanoate0.0560.0500.0870.0400.0440.0340.0700.031Ibsomyl actate0.2880.0230.0160.0270.0170.0430.0070.0160.0260.0162-Phenylethyl acetate0.0480.0180.0170.0270.0170.0430.0270.0160.0260.0162-Phenylethyl acetate0.0460.0170.0270.0170.0430.0270.0160.0260.0162-Phenylethyl acetate0.6460.5710.4740.5300.4780.5380.5240.4280.5971-Hexanol3.7125.6943.9523.0593.3782.7163.1233.4702.2683.014Octanoic acid6.2906.1115.6294.8374.6764.4854.2593.9584.7214.015Decanoic acid0.6570.4660.5750.7300.4780.5380.5240.4280.5971-Hexanol0.7781.1260.8660.5750.7300.5180.7220.6670.0750.111Linaloal*3.3291.0400.0000.8860.3274.9231.9650.6171.3790.829z*-Brenken-1-010.0890.1	Ethyl isovalerate	0.066	0.080	0.109	0.098	0.109	0.048	0.056	0.085	0.083	0.104
Ethyl lactate16.921.119.214.317.518.425.823.616.630.0Ethyl octanoate0.5660.5200.4680.5110.4080.3120.3530.3020.3590.310Ishyl decanoate0.0860.0500.0870.0400.0490.0440.0340.0700.031Isoamyl acetate0.2580.2030.1360.1380.1280.1830.1630.0930.0950.109Isoamyl acetate0.2580.2030.1360.1380.1280.1830.1630.0930.0950.109Isoamyl acetate0.2580.2030.1360.1380.1270.0170.0430.0270.0160.0260.016Isoamyl acetate0.2593.9523.0593.3782.7163.1233.4702.2683.014Octanoic acid0.5570.4660.5770.4740.5300.5180.7220.6730.4010.642trans-3-bexen-1-ol0.1130.1870.1270.0780.1140.0280.0590.0330.0190.043circs-3-bexen-1-ol0.0130.1870.1720.0690.0910.0650.0750.111Linaloal*3.3291.0400.0000.8860.3274.9231.9650.6171.3790.829extry lachoh0.0790.1040.0770.0770.0720.6690.9910.6650.6750.111Linaloal* </td <td>Ethyl hexanoate</td> <td>0.536</td> <td>0.492</td> <td>0.462</td> <td>0.487</td> <td>0.431</td> <td>0.358</td> <td>0.389</td> <td>0.353</td> <td>0.379</td> <td>0.374</td>	Ethyl hexanoate	0.536	0.492	0.462	0.487	0.431	0.358	0.389	0.353	0.379	0.374
Ethyl octanoate 0.566 0.520 0.468 0.511 0.408 0.312 0.353 0.302 0.359 0.310 Ethyl octanoate 0.080 0.066 0.050 0.087 0.040 0.049 0.044 0.031 0.031 0.031 Stoamyl acctate 0.258 0.203 0.136 0.138 0.128 0.183 0.163 0.027 0.016 0.026 0.016 Sovaleric acid 1.315 1.459 1.271 1.249 1.121 1.251 1.094 1.198 1.211 2.312 Hexanoic acid 0.657 0.466 0.577 0.474 0.530 0.478 0.538 0.524 0.428 0.597 1-Hexanol 0.778 1.126 0.866 0.575 0.730 0.518 0.722 0.673 0.401 0.642 trans-3-bexen-1-ol 0.113 0.187 0.127 0.078 0.114 0.028 0.059 0.033 0.019 0.043 cis-3-bexen-1-ol 0.089	Ethyl lactate	16.9	21.1	19.2	14.3	17.5	18.4	25.8	23.6	16.6	30.0
Erbyl decanoate DORG <thdorg< th=""> DORG DORG</thdorg<>	Ethyl octanoate	0.566	0.520	0.468	0.511	0.408	0.312	0.353	0.302	0.359	0.310
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	Ethyl decanoate	0.080	0.066	0.050	0.087	0.040	0.049	0.044	0.034	0.070	0.031
$ \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c}$	Isoamyl acetate	0.258	0 203	0.136	0 1 3 8	0.128	0 1 8 3	0 163	0.004	0.095	0 109
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	2-Phenylethyl acetato	0.230	0.203	0.150	0.130	0.120	0.103	0.105	0.035	0.035	0.105
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	2-1 nonyioniyi doeldle	1 215	1 450	1.271	1 2 / 0	1 1 2 1	1 251	1.027	1 100	1 211	0.010
$\begin{array}{c} \mbox{rectur}{Texahol} \mbox{actur}{actur}{actur} & 5.712 & 5.02 & 5.029 & 5.76 & 2.716 & 3.123 & 3.470 & 2.208 & 3.014 \\ 0ctanoic acid & 0.657 & 0.466 & 0.577 & 0.474 & 0.530 & 0.478 & 0.538 & 0.524 & 0.428 & 0.597 \\ 1-Hexahol & 0.778 & 1.126 & 0.866 & 0.575 & 0.730 & 0.518 & 0.722 & 0.673 & 0.401 & 0.642 \\ trams-3-hexen-1-ol & 0.013 & 0.187 & 0.127 & 0.078 & 0.114 & 0.028 & 0.059 & 0.033 & 0.019 & 0.043 \\ cis-3-hexen-1-ol & 0.089 & 0.102 & 0.062 & 0.049 & 0.058 & 0.187 & 0.275 & 0.174 & 0.129 & 0.257 \\ Benzyl alcohol & 0.079 & 0.104 & 0.077 & 0.077 & 0.072 & 0.069 & 0.091 & 0.065 & 0.075 & 0.111 \\ Linalool^* & 3.329 & 1.040 & 0.000 & 0.886 & 0.327 & 4.923 & 1.965 & 0.617 & 1.379 & 0.829 \\ a^-Terpineol^* & 4.497 & 3.294 & 3.382 & 4.802 & 3.616 & 4.716 & 4.572 & 4.048 & 5.863 & 6.048 \\ Citronellol^* & 1.368 & 0.696 & 0.625 & 0.522 & 0.402 & 1.772 & 1.343 & 0.678 & 0.878 & 1.272 \\ \gamma-Nonalactone^* & 1.659 & 1.336 & 1.544 & 1.865 & 1.672 & 1.847 & 1.644 & 1.798 & 2.193 & 1.996 \\ Vanillin^* & 1.441 & nd & 3.953 & 2.341 & 1.495 & 1.630 & nd & 3.055 & 23.27 & 31.83 \\ Methyl vanillate^* & f.61 & 1.92 & 2.53 & 5.91 & 3.26 & 5.80 & 1.38 & 2.35 & 6.42 & 6.95 \\ Ethyl vanillate^* & nd & n$	Hovanoic acid	1.515	1.439 E 604	2.052	2.050	1.121	1.201	1.094	1.190	1.211	2.312
Octation: actu 0.290 0.111 5.629 4.837 4.676 4485 4.259 3.958 4.721 4.015 Decanoic acid 0.657 0.466 0.577 0.474 0.530 0.478 0.538 0.524 0.428 0.597 1-Hexanol 0.778 1.126 0.866 0.575 0.730 0.518 0.722 0.673 0.401 0.642 trans-3-hexen-1-ol 0.113 0.187 0.127 0.078 0.114 0.028 0.059 0.033 0.019 0.043 cis-3-hexen-1-ol 0.089 0.102 0.062 0.049 0.058 0.187 0.275 0.174 0.129 0.257 Benzyl alcohol 0.079 0.104 0.0077 0.072 0.069 0.091 0.065 0.075 0.111 Linalool* 3.329 1.040 0.000 0.886 0.327 4.923 1.665 0.617 1.379 0.829 citronellol* 1.368 0.666 0.625 </td <td></td> <td>3./12</td> <td>5.694</td> <td>3.952</td> <td>3.059</td> <td>3.3/8</td> <td>2./10</td> <td>5.125</td> <td>3.470</td> <td>2.268</td> <td>3.014</td>		3./12	5.694	3.952	3.059	3.3/8	2./10	5.125	3.470	2.268	3.014
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Octalioic acid	6.290	6.111	5.629	4.83/	4.0/0	4.485	4.259	3.958	4./21	4.015
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Decanoic acid	0.657	0.466	0.577	0.474	0.530	0.478	0.538	0.524	0.428	0.597
$ \begin{array}{c} trans-3-hexen-1-ol \\ cis-3-hexen-1-ol \\ cis-3-hexen-1-ol \\ cis-3-hexen-1-ol \\ cis-3-hexen-1-ol \\ 0.089 \\ 0.102 \\ 0.062 \\ 0.049 \\ 0.062 \\ 0.049 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.066 \\ 0.091 \\ 0.065 \\ 0.075 \\ 0.174 \\ 0.129 \\ 0.055 \\ 0.075 \\ 0.111 \\ 0.129 \\ 0.055 \\ 0.075 \\ 0.111 \\ 0.129 \\ 0.055 \\ 0.075 \\ 0.111 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.075 \\ 0.111 \\ 0.078 \\ 0.091 \\ 0.065 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.075 \\ 0.111 \\ 0.078 \\ 0.091 \\ 0.065 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.017 \\ 0.078 \\ 0.078 \\ 0.091 \\ 0.065 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.077 \\ 0.072 \\ 0.092 \\ 0.091 \\ 0.065 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.061 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.078 \\ 0.083$	I-Hexanol	0.778	1.126	0.866	0.575	0.730	0.518	0.722	0.673	0.401	0.642
$\begin{array}{c} crs-4 nexen-1-ol \\ crs-4 nexen-1-ol \\ benzyl alcohol \\ 0.079 \\ 0.104 \\ 0.079 \\ 0.104 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.077 \\ 0.072 \\ 0.069 \\ 0.091 \\ 0.065 \\ 0.061 \\ 0.065 \\ 0.075 \\ 0.174 \\ 0.065 \\ 0.075 \\ 0.179 \\ 0.082 \\ 0.082 \\ 0.075 \\ 0.111 \\ 0.088 \\ 0.327 \\ 4.923 \\ 1.965 \\ 0.617 \\ 1.379 \\ 0.829 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.678 \\ 0.67 \\ 0.71 \\ 0.076 \\ 0.287 \\ 0.07 \\ 0.007 \\ 0.007 \\ 0.007 \\ 0.007 \\ 0.073 \\ 0.073 \\ 0.076 \\ 0.073 \\ 0.073 \\ 0.075 \\ 0.073 \\ 0.075 \\ 0.071 \\ 0.046 \\ 0.064 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.076 \\ 0.074 \\ 0.076 \\ 0.074 \\ 0.076 \\ 0.071 \\ 0.046 \\ 0.064 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.076 \\ 0.074 \\ 0.076 \\ 0.071 \\ 0.046 \\ 0.064 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.075 \\ 0.073 \\ 0.075 \\ 0.073 \\ 0.075 \\ 0.073 \\ 0.075 \\ 0.075 \\ 0.071 \\ 0.046 \\ 0.064 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.073 \\ 0.075 \\ 0.075 \\ 0.0$	trans-3-hexen-1-ol	0.113	0.187	0.127	0.078	0.114	0.028	0.059	0.033	0.019	0.043
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	cıs-3-hexen-1-ol	0.089	0.102	0.062	0.049	0.058	0.187	0.275	0.174	0.129	0.257
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Benzyl alcohol	0.079	0.104	0.077	0.077	0.072	0.069	0.091	0.065	0.075	0.111
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	Linalool*	3.329	1.040	0.000	0.886	0.327	4.923	1.965	0.617	1.379	0.829
Citronellol*1.3680.6960.6250.5220.4021.7721.3430.6780.8781.272 γ -Butyrolactone15.5615.6211.4211.3711.9213.1215.1310.827.8318.68 γ -Nonalactone*1.6591.3361.5441.8651.6721.8471.6441.7982.1931.996Vanillin*1.441nd3.95323.4114.951.630nd3.05523.2731.83Methyl vanillate*5.611.922.535.913.265.801.382.356.426.95Ethyl vanillate*ndndndndndndndndndndAcctovanillone*5.13.23.94.64.714.49.612.316.918.32-Phenylethanol36.465.044.832.041.530.852.141.027.952.11-propanol20.518.313.919.517.824.320.619.823.322.9Isoamyl alcohols2032052022182111701741831821874-vinylguaiacol0.1900.0530.0750.1190.0760.2870.0970.1000.1660.170MalvasíaT9T18T30T9+12 MBT18 + 12 MBT9T18T30T9+12 MBT18 + 12 MBEthyl 2-methylbutyrate0.017	α-Terpineol*	4.497	3.294	3.382	4.802	3.616	4.716	4.572	4.048	5.863	6.048
γ-Butyrolactone15.5615.6211.4211.3711.9213.1215.1310.827.8318.68γ-Nonalactone*1.6591.3361.5441.8651.6721.8471.6441.7982.1931.996Vanillin*1.441nd3.95323.4114.951.630nd3.05523.2731.83Methyl vanillate*5.611.922.535.913.265.801.382.356.426.95Ethyl vanillate*ndndndndndndndndndndAcetovanillone*5.13.23.94.64.714.49.612.316.918.32-Phenylethanol36.465.044.832.041.530.852.141.027.952.11-propanol20.518.313.919.517.824.320.619.823.322.9Isobutanol21.620.818.123.319.820.921.718.723.021.2Isoamyl alcohols2032052022182111701741831821874-vinylguaiacol0.1900.0530.0750.1190.0760.2870.0970.1000.1660.170Malvasía T9T18T18 + 12 MBT9T18T30T9+12 MBT18 + 12 MB0.1510.1800.149Ethyl butyrate0.1170.1370.113 <td>Citronellol*</td> <td>1.368</td> <td>0.696</td> <td>0.625</td> <td>0.522</td> <td>0.402</td> <td>1.772</td> <td>1.343</td> <td>0.678</td> <td>0.878</td> <td>1.272</td>	Citronellol*	1.368	0.696	0.625	0.522	0.402	1.772	1.343	0.678	0.878	1.272
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	γ-Butyrolactone	15.56	15.62	11.42	11.37	11.92	13.12	15.13	10.82	7.83	18.68
Vanillin*1.441nd3.95323.4114.951.630nd3.05523.2731.83Methyl vanillate*5.611.922.535.913.265.801.382.356.426.95Ethyl vanillate*ndndndndndndndndndndndAcetovanillone*5.13.23.94.64.714.49.612.316.918.32-Phenylethanol36.465.044.832.041.530.852.141.027.952.11-propanol20.518.313.919.517.824.320.619.823.322.9Isobutanol21.620.818.123.319.820.921.718.723.021.2Isoanyl alcohols2032052022182111701741831821874-vinylguaiacol0.1900.0530.0750.1190.0760.2870.0970.1000.1660.170Malvasía T9T18T30T9+12 MBT18 + 12 MBT9T18T30T9+12 MBT18 + 12 MBEthyl butyrate0.1170.1370.1130.1540.1420.1650.1830.1510.1490.049Ethyl 2-methylbutyrate0.0400.0560.0670.0710.0710.0460.0640.0730.0730.076Ethyl sovalerate0.0400.0560.	γ-Nonalactone*	1.659	1.336	1.544	1.865	1.672	1.847	1.644	1.798	2.193	1.996
Methyl vanillate*5.611.922.535.913.265.801.382.356.426.95Ethyl vanillate*ndndndndndndndndndndndAcetovanillone*5.13.23.94.64.714.49.612.316.918.32-Phenylethanol36.465.044.832.041.530.852.141.027.952.11-propanol20.518.313.919.517.824.320.619.823.322.9Isobutanol21.620.818.123.319.820.921.718.723.021.2Isoamyl alcohols2032052022182111701741831821874-vinylguaiacol0.1900.0530.0750.1190.0760.2870.0970.1000.1660.170Malvasía T9T18T30T9+12 MBT18 + 12 MBT9T18T30T9+12 MBT18 + 12 MBEthyl butyrate0.1170.1370.1130.1540.1420.1650.1830.1510.1490.046Ethyl 2-methylbutyrate0.0400.0560.0670.0710.0760.0460.0640.0730.0730.076Ethyl 2-methylbutyrate0.2470.2580.2170.4040.5460.3610.4080.334	Vanillin*	1.441	nd	3.953	23.41	14.95	1.630	nd	3.055	23.27	31.83
Ethyl vanillate* thyl vanillate*nd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd ndnd <b< td=""><td>Methyl vanillate*</td><td>5.61</td><td>1.92</td><td>2.53</td><td>5.91</td><td>3.26</td><td>5.80</td><td>1.38</td><td>2.35</td><td>6.42</td><td>6.95</td></b<>	Methyl vanillate*	5.61	1.92	2.53	5.91	3.26	5.80	1.38	2.35	6.42	6.95
LargerIndIndIndIndIndIndIndIndIndIndIndAcctovanillone*5.13.23.94.64.714.49.612.316.918.32-Phenylethanol36.465.044.832.041.530.852.141.027.952.11-propanol20.518.313.919.517.824.320.619.823.322.9Isobutanol21.620.818.123.319.820.921.718.723.021.2Isoamyl alcohols2032052022182111701741831821874-vinylguaiacol0.1900.0530.0750.1190.0760.2870.0970.1000.1660.170Malvasía T9T18T30T9+12 MBT18 + 12 MBEthyl butyrate0.1170.1370.1130.1540.1420.1650.1830.1510.1800.149Ethyl 2-methylbutyrate0.0330.0650.0640.0550.0660.0320.0630.0610.0490.056Ethyl isovalerate0.0400.0560.0670.0710.0760.0460.0640.0730.0730.076Ethyl bexanoate0.2470.2580.2170.24040.5460.3610.4080.334	Ethyl vanillate*	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Actovaliation5.15.25.34.04.714.45.012.510.918.52-Phenylethanol 36.4 65.0 44.8 32.0 41.5 30.8 52.1 41.0 27.9 52.1 1-propanol 20.5 18.3 13.9 19.5 17.8 24.3 20.6 19.8 23.3 22.9 Isobutanol 21.6 20.8 18.1 23.3 19.8 20.9 21.7 18.7 23.0 21.2 Isoamyl alcohols 203 205 202 218 211 170 174 183 182 187 4-vinylguaiacol 0.190 0.053 0.075 0.119 0.076 0.287 0.097 0.100 0.166 0.170 MalvasíaT9T18T30T9+12 MBT18 + 12 MBT9T18T30T9+12 MBT18 + 12 MBEthyl butyrate 0.117 0.113 0.154 0.142 0.165 0.183 0.151 0.180 0.149 Ethyl 2-methylbutyrate 0.033 0.065 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.247 0.258 0.217 0.404 0.546 0.361 0.408 0.334	Acetovanillone*	5.1	3.2	30	46	47	14 4	96	12.2	160	183
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	2_Dhanylothanel	26 4	5.2 65.0	5.5 AA Q	32.0	-1.7 /115	20.0	5.0	12.J /1 0	270	52.1
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	2-michylethidhol	20.4	100.0	12.0	52.0 10 F	41.J 17.0	30.0	J2.1	41.0	27.9	JZ.1
Isooutanoi 21.6 20.8 18.1 23.3 19.8 20.9 21.7 18.7 23.0 21.2 Isoamyl alcohols 203 205 202 218 211 170 174 183 182 187 4-vinylguaiacol 0.190 0.053 0.075 0.119 0.076 0.287 0.097 0.100 0.166 0.170 Malvasía T9 T18 T30 T9+12 MB T18 + 12 MB T9 T18 T30 T9+12 MB T18 + 12 MB Ethyl butyrate 0.117 0.137 0.113 0.154 0.142 0.165 0.183 0.151 0.180 0.149 Ethyl 2-methylbutyrate 0.033 0.065 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.040 0.056 0.067 0.071 0.046 0.064 0.073 0.073 0.076 Ethyl savapate 0.247 0.258 0.217	i-propatioi	20.5	18.3	13.9	19.5	1/.ð	24.3	20.6	19.8	23.3	22.9
Isoamy alconois 203 205 202 218 211 170 174 183 182 187 4-vinylguaiacol 0.190 0.053 0.075 0.119 0.076 0.287 0.097 0.100 0.166 0.170 Malvasía T9 T18 T30 T9+12 MB T18 + 12 MB T9 T18 T30 T9+12 MB T18 + 12 MB Ethyl butyrate 0.117 0.137 0.113 0.154 0.142 0.165 0.183 0.151 0.180 0.149 Ethyl butyrate 0.033 0.065 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.040 0.056 0.067 0.071 0.071 0.046 0.064 0.073 0.073 0.076 Ethyl isovalerate 0.247 0.258 0.217 0.404 0.546 0.361 0.408 0.334	ISODUTANOI	21.6	20.8	18.1	23.3	19.8	20.9	21./	18./	23.0	21.2
4-vinyigualacoi 0.190 0.053 0.075 0.119 0.076 0.287 0.097 0.100 0.166 0.170 Malvasía T9 T18 T30 T9+12 MB T18 + 12 MB T9 T18 T30 T9+12 MB T18 + 12 MB T9 T18 T30 T9+12 MB T18 + 12 MB T9 T18 T30 T9+12 MB 0.149 0.149 0.056 0.061 0.049 0.056 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl 2-methylbutyrate 0.030 0.056 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.0404 0.056 0.067 0.071 0.0404 0.546 0.361 0.0408 0.334	Isoamyl alcohols	203	205	202	218	211	170	174	183	182	187
Malvasía T9 T18 T30 T9+12 MB T18+12 MB Garnacha-A T30 T9+12 MB T18+12 MB Ethyl butyrate 0.117 0.137 0.113 0.154 0.142 0.165 0.183 0.151 0.180 0.149 Ethyl 2-methylbutyrate 0.033 0.065 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.040 0.056 0.067 0.071 0.046 0.064 0.073 0.076 Ethyl psynopte 0.247 0.258 0.217 0.404 0.546 0.361 0.408 0.334	4-vinylguaiacol	0.190	0.053	0.075	0.119	0.076	0.287	0.097	0.100	0.166	0.170
T9 T18 T30 T9+12 MB T18 + 12 MB T9 T18 T30 T9+12 MB T18 + 12 MB Ethyl butyrate 0.117 0.137 0.113 0.154 0.142 0.165 0.183 0.151 0.180 0.149 Ethyl 2-methylbutyrate 0.033 0.065 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.040 0.056 0.067 0.071 0.071 0.0464 0.073 0.073 0.076 Ethyl bexanoate 0.247 0.258 0.217 0.404 0.546 0.361 0.408 0.334		Malvasía		ang -	mo :-		Garnacha-	-A	mo -	mo :-	
Ethyl butyrate0.1170.1370.1130.1540.1420.1650.1830.1510.1800.149Ethyl 2-methylbutyrate0.0330.0650.0640.0550.0660.0320.0630.0610.0490.056Ethyl isovalerate0.0400.0560.0670.0710.0710.0460.0640.0730.0730.076Ethyl bexanoate0.2470.2580.2270.2580.2170.4040.5460.3610.4080.334		Т9	T18	T30	T9+12 MB	T18 + 12 MB	Т9	T18	T30	T9+12 MB	T18 + 12 MB
Ethyl 2-methylbutyrate 0.033 0.065 0.064 0.055 0.066 0.032 0.063 0.061 0.049 0.056 Ethyl isovalerate 0.040 0.056 0.071 0.071 0.046 0.064 0.073 0.073 0.076 Ethyl isovalerate 0.247 0.258 0.227 0.258 0.217 0.404 0.546 0.361 0.408 0.334	Ethyl butyrate	0.117	0.137	0.113	0.154	0.142	0.165	0.183	0.151	0.180	0.149
Ethyl isovalerate 0.040 0.056 0.067 0.071 0.071 0.046 0.064 0.073 0.076 Ethyl isovalerate 0.247 0.258 0.217 0.404 0.546 0.361 0.408 0.334	Ethyl 2-methylbutyrate	0.033	0.065	0.064	0.055	0.066	0.032	0.063	0.061	0.049	0.056
Ethyl hexanoate 0.247 0.258 0.227 0.258 0.217 0.404 0.546 0.361 0.408 0.334	Etherl in the set	0.040	0.050	0.067	0.071	0.071	0.046	0.064	0.073	0.072	0.076
	Ethvi isovalerare	0.040	0 0 0 0 0	0.007	0.071	0.071	()()4()	() () ()	()())	0.073	0.070

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Table 1 (continued)

	/ 1/// 1// 1//				veruejo					
	T9	T18	T30	T9+12 MB	T18 + 12 MB	T9	T18	T30	T9+12 MB	T18 + 12 MB
Ethyl lactate	11.2	16.3	14.6	10.7	22.2	17.3	18.9	19.8	14.2	16.2
Ethyl octanoate	0.199	0.222	0.173	0.205	0.144	0.386	0.495	0.317	0.387	0.308
Ethyl decanoate	0.033	0.027	0.019	0.042	0.017	0.067	0.058	0.032	0.079	0.033
Isoamyl acetate	0.198	0.186	0.098	0.121	0.096	0.192	0.154	0.097	0.107	0.087
2-Phenylethyl acetate	0.043	0.034	0.021	0.036	0.021	0.041	0.021	0.015	0.026	0.015
Isovaleric acid	1.186	1.294	1.142	1.349	1.671	0.863	1.014	0.952	1.198	0.833
Hexanoic acid	1.711	2.942	1.984	1.563	2.637	2.919	4.536	3.146	2.329	2.525
Octanoic acid	2.402	3.454	1.428	1.974	2.743	4.081	3.903	3.508	4.255	2.053
Decanoic acid	0.326	0.316	0.370	0.288	0.469	0.469	0.450	0.455	0.388	0.436
1-Hexanol	0.601	0.872	0.716	0.450	0.713	0.738	0.996	0.826	0.504	0.592
trans-3-hexen-1-ol	0.038	0.054	0.047	0.027	0.062	0.036	0.063	0.040	0.023	0.034
cis-3-hexen-1-ol	0.100	0.123	0.072	0.061	0.109	0.127	0.121	0.088	0.068	0.074
Benzyl alcohol	0.082	0.101	0.081	0.085	0.144	0.095	0.112	0.087	0.094	0.082
Linalool*	6.894	4.182	1.774	3.679	1.830	4.834	1.533	0.516	1.255	0.524
α-Terpineol*	7 584	8 489	6412	10 466	9 896	5 241	5 606	3 992	6 564	4 483
Citronellol*	2.089	1 516	0.891	1 418	1 596	1 840	1 079	0.535	0.735	0.621
v-Butvrolactone	13.65	12 39	1071	12.44	17.72	11 57	12.96	9.81	7.37	9.85
γ-Nonalactone*	1 896	1 450	1 782	2.448	1953	2 418	2.073	2,227	2,869	2,495
Vanillin*	1 276	nd	3 516	20	1914	0.867	nd 2.075	4 983	2.005	20.00
Methyl vanillate*	10.99	5 76	6.09	1240	13.62	19 39	8 49	11 18	27.63	14 79
Fthyl yanillate*	nd	0.70 nd	nd	12.40 nd	nd	3 44	1/12	2 70	6.01	3.87
Acetovanillono*	17.0	07	12.0	175	10.0	35 0	20.0	2.79	40.4	30.3
2 Dhanylathanal	17.0	9.7	12.9	17.5	19.9	26.1	20.0	27.4	40.4	30.5 20.6
	42.4	/3.0	52.9	38.4	09.0	30.1	55.6	46.2	32.3	39.0
I-propanoi	11.9	9.4	7.1	11.5	10.7	22.5	19.1	19.3	20.3	21.0
Isobutanol	15.1	14.5	11./	17.2	14.5	19.6	18.5	17.8	19.2	18./
Isoamyl alcohols	130	137	130	146	150	165	171	182	157	175
4-vinylguaiacol	0.202	0.077	0.103	0.195	0.169	0.078	0.051	0.078	0.122	0.090
	Garnacha-	-B				Prieto Picu	ıdo			
	T9	T18	T30	T9+12 MB	T18 + 12 MB	T9	T18	T30	T9+12 MB	T18 + 12 MB
Ethyl butyrate	0.142	0.103	0.134	0.161	0.153	0.184	0.178	0.170	0.189	0.173
Ethyl 2-methylbutyrate	0.023	0.026	0.046	0.038	0.049	0.026	0.051	0.050	0.041	0.052
Ethyl isovalerate	0.034	0.028	0.058	0.059	0.064	0.039	0.054	0.065	0.067	0.072
Ethyl hexanoate	0.344	0.299	0.315	0.375	0.306	0.399	0.603	0.369	0.411	0.363
Ethyl lactate	13.4	16.0	17.7	12.3	15.4	23.6	27.1	27.7	20.5	24.3
Ethyl octanoate	0.326	0.240	0.298	0.370	0.293	0.390	0.579	0.349	0.403	0.344
Ethyl decanoate	0.054	0.034	0.031	0.064	0.031	0.054	0.051	0.032	0.068	0.032
Isoamyl acetate	0.170	0.081	0.081	0.096	0.082	0.596	0.537	0.202	0.268	0.188
2-Phenylethyl acetate	0.039	0.020	0.015	0.026	0.015	0.136	0.083	0.048	0.088	0.046
Isovaleric acid	0.905	1.229	0.873	1.304	0.771	1.283	1.600	1.154	1.581	1.050
Hexanoic acid	2.456	3.643	2.910	2.016	2.517	3.251	4.398	3.381	2.565	2.937
Octanoic acid	4.276	4.461	3.578	3,699	2,345	4.451	4.430	3.694	4,109	2.114
Decanoic acid	0.458	0.684	0 456	0.376	0.433	0 449	0 575	0 457	0.387	0.421
1-Hexanol	0.673	0.846	0.758	0.459	0.599	0 475	0 854	0 519	0.365	0.428
trans-3-hexen-1-ol	0.034	0.048	0.041	0.020	0.035	0.055	0.098	0.057	0.037	0.049
cis-3-hexen-1-ol	0 115	0.112	0.085	0.059	0.076	0.109	0.116	0.078	0.066	0.068
Benzyl alcohol	0.094	0.112	0.003	0.033	0.076	0.105	0.110	0.078	0.000	0.000
Linalool*	1 0.034 1 0.1 Q	1 506	0.092	1 571	0.030	6.675	2 2 2 1	1 707	3 5 2 1	1 786
a-Ternineol*	-1.340 5 05 1	5 200	4.610	5.012	4.622	5 619	5.301	5,627	7 257	5 012
Citropollol*	2.021	1 217	4.010	0.914	4.033	J.010 2 E 10	0.240	1 200	1.207	1 212
v Puturolactore	2.028	1.21/	0.700	0.014	0.750	2.510	2.200	1.300	1.600	1.212
γ-Dulyrolacione	11.10	12.63	10.21	9.91	10.73	14.99	10.49	10.67	14.0/	10.97
γ-inolialactone" Venillin*	2.28/	1.876	2.141	2./40	2.301	1.990	1./2/	1.839	2.496	2.016
vanillin	1.711	nd	3.690	25./1	27.27	2.409	nd	2.366	23.07	20.33
Methyl vanillate*	23.13	13.65	12.82	24.09	14.83	14.03	5.33	7.68	11.00	8.61
Ethyl vanillate*	3.24	1.70	2.91	5.49	3.77	16.53	7.83	11.03	16.15	14.45
Acetovanillone*	35.3	19.7	28.0	38.4	31.5	16.7	9.7	12.5	11.2	14.1
2-Phenylethanol	33.7	67.7	47.5	34.6	42.6	42.5	68.1	50.5	41.1	44.5
	20.7	15.7	18.1	19.4	19.1	33.5	33.9	30.2	34.1	34.4
I-propanol			10.4	10.4	107	20.2	30.3	261	27.8	28.3
I-propanol Isobutanol	18.3	15.8	18.4	19.4	10.7	23.2	50.5	2011	2110	2015
I-propanol Isobutanol Isoamyl alcohols	18.3 155	15.8 144	18.4 179	19.4	172	204	217	215	188	211

nd: no detected.

Gallardo-Chacón et al., 2010; Riu-Aumatell et al., 2006), and it can last from a minimum of 9 months to years.

Foam is the characteristic that differentiates sparkling wines from still wines, being the first sensory attribute that tasters and consumers perceive and that determines the final quality of sparkling wines (Buxaderas & López-Tamames, 2012). The foaming properties mainly depend on the chemical composition of wines (Gallart, Lopez-Tamames, Suberbiola, & Buxaderas, 2002; LópezBarajas, López-Tamames, Buxaderas, Tomás, & De La Torre, 1999; Moreno-Arribas, Puevo, Nieto, Martín-Álvarez, & Polo, 2000), and different factors involved in wine composition will have an effect on foam quality, among them the grape variety used (Andrés-Lacueva et al., 1996; Cilindre, Liger-Belair, Villaume, Jeandet, & Marchal, 2010; Girbau-Solà, López-Barajas, López-Tamames, & Buxaderas, 2002; López-Barajas, López-Tamames, Buxaderas, & De La Torre, 1998). In addition, the foamability of sparkling wines can

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Multifactor analysis of variance carried out considering all the data of all the spar-

	Grape v	ariety	Aging ti	me	Grape v \times aging	ariety time
	F-ratio	P-value	F-ratio	P-value	F-ratio	P-value
Ethyl butyrate	49.8	0.000	15.0	0.000	11.8	0.000
Ethyl 2-methylbutyrate	141	0.000	287	0.000	9.1	0.000
Ethyl isovalerate	144	0.000	287	0.000	7.8	0.000
Ethyl hexanoate	243	0.000	71.1	0.000	10.6	0.000
Ethyl lactate	147	0.000	136	0.000	10.3	0.000
Ethyl octanoate	450	0.000	130	0.000	16.7	0.000
Ethyl decanoate	143	0.000	550	0.000	7.3	0.000
Isoamyl acetate	537	0.000	691	0.000	53.7	0.000
2-Phenylethyl acetate	570	0.000	602	0.000	28.3	0.000
Isovaleric acid	30.8	0.000	15.6	0.000	14.5	0.000
Hexanoic acid	181	0.000	186	0.000	10.8	0.000
Octanoic acid	349	0.000	128	0.000	10.8	0.000
Decanoic acid	7.9	0.000	6.6	0.000	1.4	0.142
1-Hexanol	164	0.000	401	0.000	8.2	0.000
trans-3-hexen-1-ol	623	0.000	315	0.000	15.4	0.000
cis-3-hexen-1-ol	801	0.000	481	0.000	35.2	0.000
Benzyl alcohol	62.3	0.000	65.4	0.000	18.0	0.000
Linalool	420	0.000	2242	0.000	21.7	0.000
α-Terpineol	391	0.000	85.3	0.000	8.2	0.000
Citronellol	357	0.000	870	0.000	20.7	0.000
γ-Butyrolactone	55.3	0.000	112	0.000	23.3	0.000
γ-Nonalactone	166	0.000	208	0.000	2.4	0.001
Vanillin	29.1	0.000	1397	0.000	17.0	0.000
Methyl vanillate	436	0.000	325	0.000	22.0	0.000
Ethyl vanillate	2215	0.000	108	0.000	39.8	0.000
Acetovanillone	2231	0.000	474	0.000	49.8	0.000
2-Phenylethanol	66.4	0.000	463	0.000	10.6	0.000
1-propanol	881	0.000	107	0.000	5.4	0.000
Isobutanol	547	0.000	71.3	0.000	8.4	0.000
Isoamyl alcohols	490	0.000	35.5	0.000	13.9	0.000
A_vinvlguaiacol	440	0.000	513	0.000	138	0.000

Values in bold showed statistically significant differences in each compound and factor considered (P-values < 0.05).

be influenced by the aging time on lees (Andres-Lacueva, Lamuela-Ravento;s, Buxaderas, & De La Torre-Boronat, 1997; Girbau-Sola et al., 2002; Moreno-Arribas et al., 2000).

However, few studies have been found in relation to the volatile composition of sparkling wines after aging times longer than 9 months (Francioli et al., 2003; Riu-Aumatell et al., 2006; Torrens et al., 2010), as well as the changes in their foamability (Andrés-Lacueva et al., 1996; Girbau-Sola et al., 2002), and no one has been found in relation to the changes that can occur in sparkling wines after disgorging, i.e. without lees.

Therefore, the aim of this work was focused on the study of the influence of grape variety and aging time in contact with lees and without lees, on volatile composition and foamability of white and rosé sparkling wines. Seven different grape varieties were used and the sparkling wines were studied until 30 months of aging on lees. In addition, the sparkling wines were also analyzed after 12 months in bottle after disgorging.

2. Material and methods

2.1. Winemaking process

The grape varieties used in this study were: Verdejo and Viura from the Designation of Origin (D.O.) Rueda, Malvasía from the D.O. Toro, Albarín from the D.O. Tierra de León and Godello from the D.O. Bierzo, for the elaboration of white sparkling wines, and Prieto Picudo from the D.O. Tierra de León and Garnacha from the D.O. Cigales for rosé sparkling wines. Two different vineyards of Garnacha grape variety were used. Only Verdejo and Godello grape varieties have been used to elaborate sparkling wines, but all of them produced high quality still wines in these D.O.s.

The base wines were elaborated in the experimental winery of the Enological Station sited in Rueda (Valladolid), following the traditional white or rosé winemaking process in stainless steel tanks of 150 L. The same commercial Saccharomyces cerevisiae yeasts (IOC 18-2007, Lallemand, Spain) were used.

The sparkling wines were elaborated following the traditional or "champenoise" method, therefore the base wines, after coldstabilization (at -5 °C) and clarification (with PVPP and bentonite), were bottled and the tirage liquor was added. The tirage liquor were formed by yeast S. cerevisiae var. bayanus (0.30 g/L, IOC 18-2007 Lallemand, Spain), sucrose (23 g/L) and bentonite (0.10 g/L) (Laffort, France). After that, the bottles were kept in a cellar at a temperature of 11–13 °C and at a relative humidity of 75–85% controlled for 30 months. The pressure and residual sugars were measured periodically to control the second fermentation.

Sparkling wines were analyzed after 9, 18 and 30 months of aging on lees. These sampling points were selected according to representative aging periods of sparkling wine categories: sparkling wine (\geq 9 months), Reserve (\geq 15 months) and Great Reserve $(\geq 30 \text{ months})$. In addition, the sparkling wines aged on lees for 9 and 18 months were also analyzed after 12 months in bottle after disgorging. Before the analyses, the wines were riddled and disgorged. Brut Nature sparkling wines were obtained, i.e. no expedition liqueur was added.

Therefore, eight sparkling wines were elaborated and were analyzed in five aging times.

2.2. Chemical reagents

The volatile compound standards were purchased from Fluka (Buchs, Switzerland) (ethyl butyrate, ethyl isovalerate, ethyl hexanoate, ethyl octanoate, ethyl lactate, 2-phenylethyl acetate, isobutanol, benzyl alcohol, 2-methyl-1-butanol, 3-methyl-1-butanol, 1-propanol, 2-phenylethanol, 1-hexanol, cis-3-hexenol, hexanoic acid, octanoic acid, decanoic acid, isovaleric acid, γ -butyrolactone, citronellol, α-terpineol, vanillin); Sigma–Aldrich (Steinheim, Germany) (ethyl 2-methylbutyrate, ethyl decanoate, isoamyl acetate, *trans*-3-hexenol, γ -nonalactone, acetovanillone, linalool, methyl octanoate); and Lancaster (Strasbourg, France) (methyl vanillate, ethyl vanillate, 4-vinylguaiacol, 3,4-dimethylphenol).

The remaining reagents were supplied by Panreac (Madrid, Spain). Water Milli-Q was obtained via a Millipore system (Bedford, MA).

2.3. Analysis of the volatile compounds

Volatile compounds were extracted by liquid-liquid extraction following the method developed by Rodríguez-Bencomo, Ortega-Heras, & Pérez-Magariño (2010). Two hundred and 50 mL of wine, 5 mL of dichloromethane, and 75 µL of a mixture of two internal IS standards (550 mg/L of methyl octanoate, and 450 mg/L of 3,4-dimethylphenol) were added to a flask. The extraction was carried out for 3 h with continuous stirring (150 rpm) in an orbital shaker. Chromatographic analyses were performed with a HP-6890N GC coupled to a HP-5973 inert MS detector equipped with a Quadrex 007CWBTR capillary column (60 m length, 0.25 mm i.d., and 0.25 µm film thickness), following the chromatographic conditions established by Rodríguez-Bencomo et al. (2010).

Quantification was carried out following the internal standard quantification method. Quantitative data of the relative areas (absolute areas/internal standard area) were subsequently interpolated in the calibration graphs built from results of pure reference S. Pérez-Magariño et al. / LWT - Food Science and Technology xxx (2014) 1-10

Table 3

Factor loadings after varimax rotation of the sparking wines aged on lees.

Compounds	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5	Factor 6	Factor 7	Factor
Ethyl butyrate	0.547		0.563		-0.301			
Ethyl 2-methylbutyrate		-0.714		0.447		-0.400		
Ethyl isovalerate	0.274	-0.793		0.299	-0.281			
Ethyl hexanoate	0.887		0.333					
Ethyl lactate		-0.270	0.809					0.320
Ethyl octanoate	0.907		0.324					
Ethyl decanoate	0.841	0.399						
Isoamyl acetate	0.318	0.625	0.565					
2-Phenylethyl acetate		0.745	0.572					
Isovaleric acid				0.807				
Hexanoic acid	0.742	-0.291	0.267		0.317			
Octanoic acid	0.841					0.290		
Decanoic acid	0.435						-0.415	
1-Hexanol	0.632	-0.373			0.547			
trans-3-hexen-1-ol	0.761	-0.256		0.302	0.281			
cis-3-hexen-1-ol								0.954
Benzyl alcohol	0.434				0.800			
Linalool		0.892					0.301	
α-Terpineol							0.894	
Citronellol		0.942						
γ-Butvrolactone	0.263	0.561		0.585				
γ-Nonalactone				-0.866			0.325	
Vanillin	-0.275	-0.338			-0.556			-0.449
Methyl vanillate		0.453		-0.829				
Ethyl vanillate		0.396	0.751			-0.312		-0.277
Acetovanillone	-0.279			-0.857				
2-Phenylethanol				0.403	0.733	-0.419		
1-propanol	0.378	0.296	0.784			0.287		
Isobutanol	0.340	0.321	0.838					
Isoamvl alcohols	0.487		0.733					
4-vinvlguaiacol						0.883		
Eigenvalue	8.83	6.53	3.34	3.15	2.17	1.44	1.40	1.05
Cumulative variance (%)	28.5	49.6	60.3	70.5	77.5	82.1	86.6	90.0

Loadings lower than absolute values of 0.250 are not shown.

Values in bold indicate the highest weight of each compound in each factor.

compounds (Pérez-Magariño, Ortega-Heras, Martínez-Lapuente, Guadalupe, & Ayestarán, 2013).

Three bottles of each varietal sparkling wine at each sampling time were analyzed, and one extraction for each bottle was carried out.

2.4. Measurement of foaming properties by instrumental method

The foam measurement of sparkling wines was carried out using the Mosalux procedure (Maujean, Poinsaut, Dantan, Brissonet, & Cossiez, 1990). This equipment consists in a glass cylinder with a glass frit in the bottom. This cylinder was filled with 100 mL of wine and CO₂ was injected through the glass frit at a rate of 7 L/h under a constant pressure of 1 bar for 15 min. Then the gas injection was stopped.

Three parameters were measured: HM (expressed in mm) was the maximum height reached by the foam after CO_2 injection that represents the foamability; HS (expressed in mm) was the foam stability height during CO_2 injection that represents the persistence of the foam collar; and TS (expressed in sec) was the foam stability time until all bubbles collapse after the CO_2 flow has stopped.

Three bottles of each varietal sparkling wine at each sampling time were analyzed, and three measurements for each bottle were carried out.

2.5. Statistical analyses

Multifactor analyses of variance were performed. Factor analysis was applied in order to study the association of volatile compounds and to determine similarities or differences between wines by grape variety or by aging time. Varimax rotation criterion was performed and only factors with eigenvalues greater than 1 were selected. Foam parameters were treated applying the variance analysis (ANOVA), and the Least Significant Difference test at significant level of p < 0.05. These statistical analyses were carried out using the Statgraphics Plus 5.0 statistical package.

3. Results and discussion

3.1. Volatile compounds of sparkling wines during aging on lees and aging in bottle after disgorging

Table 1 shows the data of volatile compounds of sparkling wines at the different aging times studied. Due to the high number of data, initially, multifactor analysis was carried out with all the data. Table 2 shows the effects of grape variety, aging time, and the interaction of grape variety-aging time for each compound. In general, it can be observed that there are strong grape variety and aging time effects on all volatile compounds evaluated.

Therefore, factorial analyses were carried out in order to study the influence of grape variety and aging time on the volatile profile of the sparkling wines elaborated. Two different factorial analyses were carried out, one considering only the sparkling wines aged on lees and other considering the wines aged on lees and those aged in bottle after disgorging (without lees).

The factorial analysis carried out with the sparkling wines only aged on lees selected eight factors with an eigenvalue greater than 1, which explained the 90.0% of the total variance. However, the first six factors were enough to explain more than 80% of total variability. Table 3 shows the loadings of each compound in each S. Pérez-Magariño et al. / LWT - Food Science and Technology xxx (2014) 1–10

one of the selected factors, as well as the eigenvalue and the cumulative variance of each factor. The compounds with higher loading values contribute most significantly to the explanatory meaning of the factors (marked in bold).

Fig. 1a shows the distribution of the different sparkling wines studied in the plane defined by the first two factors, which explained the 49.6% of the total variance. As it can be seen in this figure, the variables mainly associated with factor 2 allow differentiating the sparkling wines by the aging time on lees. The sparkling wines aged on lees for 9 months were sited in the positive zone of factor 2, and along the aging (18 and 30 months), the values of factor 2 decreased. This fact was due to the increase of ethyl esters of branched-chain fatty acids, and to the decrease of higher alcohol acetates and terpenes (mainly linalool and citronellol), compounds associated to factor 2 (Table 3). Rodríguez-Bencomo et al. (2010) also observed these changes in still red wine aged on lees, and Hidalgo et al. (2004) also found an increase of some ethyl esters of Garnacha rosé sparkling wines during the aging on lees for 9 months. In addition, Francioli et al. (2003) and Riu-Aumatell et al. (2006) observed lower concentrations of higher alcohol acetates and higher of TDN (1,1,6-trimethyl-l,2-dihydronaphthalene), vitispirane and diethylsuccinate in Cava wines aged on lees for long period of time (more than 20 months) than in young ones (9 months of aging). Torrens et al. (2010) also found a decrease of higher alcohol acetates along the aging of Cava wines (until 24 months of aging on lees). Riu-Aumatell et al. (2006) also asserted that these compounds could be used as age markers.

In general, wines aged on lees for 9 and 18 months showed slightly higher values of factor 3 than wines aged for 30 months in each grape variety (Fig. 1b), which was mainly due to the increase of

ethyl lactate and isoamyl alcohols. These results agree with those obtained by Riu-Aumatell et al. (2006) and Torrens et al. (2010) in white and rosé Cava wines.

Considering the results obtained by Gallardo-Chacón et al. (2009 and 2010), the decrease of terpenes and ethyl esters of long-chain fatty acids could be due to their adsorption on the yeast cell walls, since they concluded that the most hydrophobic compounds were more retained by the lees surface.

On the other hand, the plane defined by the factors 1 and 3, which explained the 39.3% of the data variability, allows differentiating the wines by grape variety (Fig. 1b). Prieto Picudo sparkling wines showed the highest values of factor 3, which indicates that these wines had the highest concentrations of volatile compounds associated with factor 3, ethyl lactate, ethyl vanillate, and higher alcohols (Table 3). Albarín, Verdejo and Godello sparkling wines were the richest in volatile compounds associated with factor 1, ethyl esters of straight-chain fatty acids, fatty acids, 1-hexanol and trans-3-hexen-1-ol (Table 3). On the contrary, Malvasía sparkling wines were the poorest in volatile compounds associated with factors 1 and 3, followed by Viura and Garnacha sparkling wines. Higher alcohols, mainly 1-propanol and isoamyl alcohols, were also selected as volatile compounds to differentiate among varietal wines in different studies carried out in still wines (Pozo-Bayón, Pueyo, Martín-Álvarez, & Polo, 2001; Ortega-Heras, González-Huerta, Herrera, & González-Sanjosé, 2004; Tredoux et al., 2008).

Besides, there are other compounds, such as higher alcohol acetates and terpenes that were influenced by aging time, as it has been previously commented, but also they are influenced by grape variety (data not shown). In this way, Ferreira, López, & Cacho, 2000 concluded that the volatile compounds derived from yeast amino



Fig. 1. Distribution of the sparkling wines aged on lees in the plane defined by (a) factor 1 and 2, and (b) factor 1 and 3; and of the sparkling wines aged on lees and in bottle after disgorging in the plane defined by (c) factor 1 and 2, and (d) factor 1 and 3. ● nine months of aging on lees, ▲ eighteen months of aging on lees, ■ thirty months of aging on lees, ○ nine months of aging on lees + twelve months of aging in bottle after disgorging, ○ eighteen months of aging on lees + twelve months of aging in bottle after disgorging. ● Albarín ● Verdejo ● Godello ● Viura ● Malvasía ● Garnacha-B ● Prieto Picudo.

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Table 4

Factor loadings after varimax rotation of the sparking wines aged on lees and in bottle after disgorging.

Compounds	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5	Factor 6	Factor 7	Factor 8
Ethyl butyrate		0.704					0.355	
Ethyl 2-methylbutyrate			-0.695	-0.416	0.308			-0.324
Ethyl isovalerate			-0.842					
Ethyl hexanoate	0.668	0.487				0.459		
Ethyl lactate	0.357	0.514			0.488	-0.489		
Ethyl octanoate	0.610	0.498				0.541		
Ethyl decanoate						0.871		
Isoamyl acetate	0.275	0.534	0.668					
2-Phenylethyl acetate		0.574	0.728					
Isovaleric acid					0.903			
Hexanoic acid	0.855	0.250		-0.257				
Octanoic acid	0.736					0.382		0.348
Decanoic acid	0.508				0.281		-0.509	
1-Hexanol	0.927							
trans-3-hexen-1-ol	0.813			-0.385				
cis-3-hexen-1-ol	0.287				0.328	-0.476		0.452
Benzvl alcohol	0.650			0.266	0.463			
Linalool			0.899					
α-Terpineol							0.876	
Citronellol			0.895		0.319			
γ-Butvrolactone			0.266	-0.390	0.712			
γ-Nonalactone				0.739			0.422	
Vanillin	-0.592		-0.657					
Methyl vanillate				0.887				
Ethyl vanillate	-0.362	0.638	0.323					-0.462
Acetovanillone	-0.328			0.832				
2-Phenylethanol	0.485				0.392	-0.449		-0.442
1-propanol		0.895						
Isobutanol		0.904						
Isoamyl alcohols		0.749	-0.253	-0.364			-0.297	
4-vinvlguaiacol								0.846
Eigenvalue	7.83	5.24	4.56	3.09	2.20	1.77	1.51	1.00
Cumulative variance (%)	25.2	42.1	56.8	66.8	73.9	79.6	84.5	87.7
cannanacite tarianee (70)	20.2		20.0	00.0			0.10	57.1

Loadings lower than absolute values of 0.250 are not shown.

Values in bold indicate the highest weight of each compound in each factor.

acid metabolism (higher alcohols, ethyl esters of isoacids and higher alcohol acetates) are the most important to differentiate wines by grape variety. *Prieto Picudo* sparkling wines presented the highest concentrations of higher alcohol acetates, which agrees with the higher concentrations in amino acids of this varietal wine (Pérez-Magariño et al., 2013), and terpenes, with the exception of α -terpineol. This fact corroborate the results of the study carried out by Álvarez-Pérez et al. (2012), who found that rosé wines from *Prieto Picudo* have a complex aromatic profile with a relatively high concentrations of ethyl esters and terpenes.

Therefore, the sparkling wines from *Albarín*, *Verdejo*, *Godello* and *Prieto Picudo* have in general higher concentrations of most of the volatile compounds quantified than the rest of the wines studied, especially of ethyl esters and higher alcohol acetates, compounds that contribute to the fruity aroma of wines (Ferreira, Fernández, & Cacho, 1996).

The results of factorial analysis carried out with the wines aged only on lees (for 9 and 18 months) and those aged on lees and bottle (for 9 and 18 months aged on lees and 12 months aged in bottle after disgorging) are shown in Table 4.

The distribution of the different sparkling wines studied in the plane defined by the first two factors, which explained the 42.1% of the total variance, permits to observe that the varietal characteristics of each wine were maintained both during the aging on lees and during the latter aging in bottle (without lees), as it is shown in Fig. 1c. The compounds associated with these factors are the responsible for the varietal differences of the wines, which were in general the same that those previously commented in the factorial analysis carried out with the sparkling wines aged only on lees.

On the other hand, the plane defined by the factors 1 and 3 that explained the 39.9% of the total variance, allows differentiating the sparkling wines aged on lees from those with additional aging in bottle (without lees), being the latter sited on the bottom of the plane (Fig. 1d). This means that the compounds associated positively with the factor 1 and 3, ethyl esters of straight-chain fatty acids, higher alcohol acetates, fatty acids, C6 alcohols and terpenes (mainly linalool and citronellol), decreased during the aging in bottle, while those associated negatively, ethyl esters of branchedchain fatty acids and vanillin increased (Table 4). Some of the changes observed during the aging in bottle of the sparkling wines agree with those found during the bottle aging of still wines (Díaz-Maroto, Schneider, & Baumes, 2005; Pérez-Coello, González-Viñas, García-Romero, Díaz-Maroto, & Cabezudo, 2003).

3.2. Foaming properties of sparkling wines during aging on lees and aging in bottle after disgorging

The mean values of the foam instrumental parameters of the sparkling wines during the aging on lees are shown in Table 5. After 9 months of aging on lees, *Verdejo* and *Prieto Picudo* sparkling wines presented the highest values of foam maximum height (HM), foam stability height (HS), and foam stability time (TS), followed by *Albarín* wines.

No studies have been reported in the literature focus on the foamability of sparkling wines elaborated from these grape varieties. Only some works have been found that study the foam properties of Cava or Champagne wines evaluated by Mosalux method (Andres-Lacueva et al., 1996, 1997; Girbau-Solà et al., 2002; Vanrell et al., 2007). Andres-Lacueva et al. (1996) and Vanrell et al. (2007) found that *Chardonnay* Cava wines showed higher HM values and lower TS values than *Macabeo, Xarel.lo* and *Parellada* Cava wines. The white sparkling wines aged on lees for 9 months of

Table 5

Foam parameters determined by the Mosalux method and ANOVA results of sparkling wines aged on lees.

	Grape variety	T9 ^a	T18 ^a	T30 ^a
HM ^b (mm)	Verdejo	97 a D	121 b E	129 b D
	Godello	77 a BC	113 b DE	117 b C
	Malvasía	64 a A	85 b A	104 c A
	Albarín	82 a C	112 b DE	108 b AB
	Viura	74 a B	109 b CD	103 b A
	Prieto Picudo	96 a D	120 b DE	119 b C
	Garnacha-A	72 a B	96 b A	107 b AB
	Garnacha-B	70 a AB	100 b B	113 b BC
HS ^b (mm)	Verdejo	39.3 b D	27.1 a D	47.1 c D
	Godello	13.0 a A	12.7 a B	17.8 b A
	Malvasía	13.0 b A	9.6 a A	15.3 c A
	Albarín	19.8 ab B	16.7 a C	24.1 b B
	Viura	13.7 a A	12.1 a B	16.4 b A
	Prieto Picudo	30.0 a C	30.8 a E	36.7 b C
	Garnacha-A	14.0 b A	11.6 a B	15.3 b A
	Garnacha-B	13.6 b A	11.2 a B	15.2 c A
TS ^b (sec)	Verdejo	132 a C	212 b D	205 b C
	Godello	5.8 a A	6.9 b A	7.8 c A
	Malvasía	5.5 b A	4.7 a A	7.0 c A
	Albarín	8.0 a A	32.8 b B	11.3 a A
	Viura	5.6 a A	6.7 b A	7.6 c A
	Prieto Picudo	101 a B	162 b C	156 b B
	Garnacha-A	6.3 a A	7.8 b A	7.3 b A
	Garnacha-B	5.9 a A	6.6 ab A	7.2 b A

Values with different small letters in each grape variety and each parameter or with different capital letters in each aging time and each parameter indicate statistically significant differences at p < 0.05.

^a T9, T18, T30: nine, eighteen, thirty months of aging on lees.

^b HM: foam maximum height; HS: foam stability height; TS: foam stability time.

this study showed mean values of HM similar to those of *Chardonnay* Cava wines, with the exception of *Malvasía* sparkling wines; and *Verdejo* sparkling wines showed TS values similar than those obtained in *Chardonnay* Cava wines (Andres-Lacueva et al., 1996; Vanrell et al., 2007).

Andres-Lacueva et al. (1996) did not showed differences in HS values by grape variety, while Vanrell et al. (2007) found that sparkling wines from *Chardonnay* showed the highest HS values. Taking into account the data obtained in this study, the HS values of *Verdejo* sparkling wines were also more similar to those of *Chardonnay* sparkling wines (Vanrell et al., 2007).

For red grape varieties, *Prieto Picudo* sparkling wines showed similar foam characteristics (HM and HS) than *Pinot Noir* (Vanrell et al., 2007) and *Trepat* (Girbau-Solà et al., 2002), red grape varieties traditionally used in sparkling wine elaboration.

Considering the aging time, the HM values of the sparkling wines increased until the 18 months, keeping constant until the 30 months of aging, with the exception of sparkling wines from Malvasía that continue increasing until 30 months (Table 5). On the other hand, HS values maintained constant or slightly decreased until the 18 months of aging on lees and increased after 30 months of aging. TS values also increased with the aging time, and some differences among wines were found. Sparkling wines from Verdejo, Albarín, Prieto Picudo and Garnacha-A presented their maximum TS values at 18 months after the aging on lees, while the other wines obtained their maximum values at 30 months. Although some differences were detected in the evolution of the foam parameters of the sparkling wines depending on the grape variety, it can be pointed out that in general, the quality and stability of the foam of all the sparkling wines increased over the aging on lees or were maintained stable for 30 months of aging on lees. However, Andres-Lacueva et al. (1996 and 1997) and Moreno-Arribas et al. (2000) found that the foaming properties of white Cavas depended on the aging time. They observed an increase in foamability and stability of foam at 18 months of aging on lees, but

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Foam parameters determined by the Mosalux method and ANOVA results of sparkling wines aged on lees and in bottle after disgorging.

	Grape variety	T9 ^a	T18 ^a	T9 $+12 \text{ MB}^{a}$	$T18 + 12 \ \text{MB}^{\text{a}}$
HM ^b	Verdejo	97 a D	121 b E	117 b B	119 b B
(mm)	Godello	77 a BC	113 d DE	90 b A	103 c A
	Malvasía	64 a A	85 b A	85 b A	100 c A
	Albarín	82 a C	112 bc DE	127 d C	119 cd B
	Viura	74 a B	109 b CD	111 b B	105 b A
	Prieto Picudo	96 a D	120 bc DE	128 c C	115 b B
	Garnacha-A	72 a B	96 b AB	124 d BC	106 c A
	Garnacha-B	70 a AB	100 b BC	127 c C	100 b A
HS ^b	Verdejo	39.3 b D	27.1 a D	25.4 a E	37.0 b B
(mm)	Godello	13.0 a A	12.7 a B	11.9 a AB	15.8 b A
	Malvasía	13.0 b A	9.6 a A	10.9 a A	17.3 c A
	Albarín	19.8 a B	16.7 a C	18.7 a D	34.0 b B
	Viura	13.7 a A	12.1 a B	12.6 a BC	16.5 b A
	Prieto Picudo	30.0 a C	30.8 ab E	32.9 b F	42.4 c C
	Garnacha-A	14.0 b A	11.6 a B	13.0 ab C	16.0 c A
	Garnacha-B	13.6 b A	11.2 a B	13.2 b C	14.7 c A
TS ^b	Verdejo	132 a C	212 b D	137 a B	200 b C
(sec)	Godello	5.8 a A	6.9 b A	6.1 a A	7.0 b A
	Malvasía	5.5 b A	4.7 a A	6.1 b A	7.3 c A
	Albarín	8.0 a A	32.8 b B	14.6 a A	33.7 b B
	Viura	5.6 a A	6.7 b A	6.6 b A	7.7 c A
	Prieto Picudo	101 a B	162 b C	176 b C	227 c D
	Garnacha-A	6.3 a A	7.8 b A	7.7 b A	7.7 b A
	Garnacha-B	5.9 a A	6.6 ab A	7.3 b A	6.8 b A

Values with different small letters in each grape variety and each parameter or with different capital letters in each aging time and each parameter indicate statistically significant differences at p < 0.05.

^a T9, T18: nine, eighteen months of aging on lees; 12 MB: twelve months in bottle after disgorging.

^b HM: foam maximum height; HS: foam stability height; TS: foam stability time.

after this time the sparkling wines showed a decrease in foamability and an increase in foam persistence, and concluded that the optimum time of aging for the best and most stable foam appears to be 18 months. On the other hand, these authors suggested that the increase in foamability could be due to autolysis of the yeast, in agreement with the results obtained by Maujean et al. (1990). Yeast autolysis is a slow natural process that is characterized by the hydrolysis of intracellular polymers by yeast enzymes activated after cell death, and the release of several compounds from cytoplasm and from cell wall that gradually occurs over the aging time (Alexandre and Guilloux-Benatier, 2006). Some of the compounds that stabilize the foam were released from the yeasts, such as polysaccharides, although their influence on foamability will depend on the type and molecular weight of each polysaccharide (Martínez-Lapuente, Guadalupe, Ayestarán, Ortega-Heras, & Pérez-Magariño, 2013; Moreno-Arribas et al., 2000; Núñez, Carrascosa, González, Polo, & Martínez-Rodríguez, 2006; Vanrell et al., 2002).

Table 6 shows the foam parameters of the sparkling wines aged on lees and in bottle after disgorging, and some differences were observed depending on the aging time on lees. In the sparkling wines aged on lees for 9 months, the HM parameter increased during the aging in bottle (without lees), although the HS values remained relatively constant. On the contrary, the sparkling wines aged on lees for 18 months maintained the HM values during the aging in bottle but increased their foam stability height (HS). The aging time in bottle after the aging on lees of the studied sparkling wines did not have a clear influence on the foam stability time (Table 6), being the TS values maintained constant or slightly increased, with the exception of the TS values of Prieto Picudo sparkling wines that increased between 40 and 70%, in both type of wines. Therefore, the aging time on lees and in bottle after disgorging did not reduce the foam characteristics of the sparkling wines in the period of time studied.

The varietal differences in the foam characteristics were maintained over the aging time on lees and in bottle, being Verdejo and Prieto Picudo sparkling wines those with the highest values of foam parameters.

4. Conclusions

In summary, sparkling wines from Albarín, Verdejo, Godello and Prieto Picudo grape varieties were the richest in most of the volatile compounds analyzed, especially those that contribute to the fruity aroma of wines. Although some differences were observed between the sparkling wines depending on the aging time on lees, the results obtained indicate that the sparkling wines maintain their varietal characteristics even after long aging time (at least until 30 months).

Verdejo and Prieto Picudo sparkling wines presented the best foam characteristics, followed by Albarín and Godello wines. These differences are maintained over the aging time on lees and in bottle after disgorging.

The aging time on lees improved the foam instrumental parameters of sparkling wines at least until 18 or 30 months.

Considering all the results obtained, Albarín, Verdejo, Godello and Prieto Picudo were the most interesting grape varieties to elaborate sparkling wines, following the traditional or "champenoise" method. In addition, taking into account the foam data found in the bibliography, these wines have similar foam properties than high quality sparkling wines as Champagne and Cava wines.

Uncited references

Gallart et al., 2004; González-Sanjosé et al., 2008.

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