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ORIGINAL

Determining the impact of seasoning on the volatile chemical composition of the oak wood of different *Sherry Casks*® by DTD–GC–MS

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Abstract

The casks that have contained for a certain time Sherry wines are known as *Sherry Casks*® and their use and value have increased in recent years, as they confer unique organoleptic properties to the distillates and beverages that are aged inside them. During their seasoning, these casks yield certain compounds from wood into the wines, and at the same time, they retain some compounds from these Sherry wines which, in turn, are transferred to the distillates that are aged inside the casks, so that wood acts as a transfer vector between the two alcoholic beverages. A characterization of seasoned staves with three Sherry wines, Fino, Oloroso, and Pedro Ximénez, by DTD–GC–MS has been carried out in this study. Different tendencies regarding the compounds present in the wood have been observed as follows: in general, certain compounds from wood either disappeared or decreased during its seasoning, while other compounds that come from the seasoning wine either increased or appeared in the wood during the seasoning process. This fact demonstrates that during the ageing of Sherry wines these transfer certain interesting compounds into the casks wood, which are in turn transferred to the distillates.

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Introduction

The ageing of certain products in wooden casks is a widespread practice for the production of wines, vinegars or spirits, such as armagnac, cognac, brandy, whisky, rum, and tequila and even for the production of certain beers. Ageing in casks modifies and improves the organoleptic profile of these products. The casks used are typically new, but some beverages require the use of casks that have previously contained a different product. This is the case of wines, vinegars and brandies from the Sherry area, or the high-end whiskeys known as single malt whiskeys, which are aged in casks that have been previously filled with Sherry wines, either over the whole process or during its final stage.

The seasoning of wooden casks with Sherry wines has increased in recent years, mainly due to their commercialization for the ageing of products other than Sherry wines, vinegars, or brandies. This has led the Regulatory Council of the Protected Designation of Origin (PDO) Jerez-Xérès-Sherry to register *Sherry Casks*® as a trademark. This trademark covers oak casks that have exclusively contained some type of Sherry wine for at least one year. American oak (*Quercus alba*) with a medium toast treatment is the most common wood type used for the manufacturing of these *Sherry Casks*®, which are regulated under the UNE-EN ISO/IEC 17065 standard (OECCA Fundation 2023). According to the Technical Specification set out by the Regulatory Council of the "Jerez-Xérès-Sherry" PDO, which regulates this product, seasoning is "the process to which a cask with a capacity of less than 1000 L is subjected for the first time since its manufacturing, in order to condition it for a specific period of time so that it can hold, age or mature, as the case may be, quality products such as "*Jerez-Xérès-Sherry" or "Manzanilla-Sanlúcar de Barrameda" PDOs (Protected Designations of Origin) wines, "Sherry Vinegar" PDO vinegars or "Brandy de Jerez" GI (Geographical Indication) brandies, which require the use of previously seasoned casks for their production"* (Regulatory Council 2021).

According to the technical document for *Sherry Casks*® (Regulatory Council 2021), a minimum of 85% of the total volume of a barrel must be filled with wines from the PDO "Jerez-Xérès-Sherry". The seasoning wine must remain inside the barrels over the whole processing time. Once the appropriate time has passed, the casks are completely emptied and become available for the ageing of a new product. The types of Sherry wine used for cask ageing (Fino, Oloroso, Pedro Ximénez), the quality of the wine (age), as well as the length of time that the wine remains in the wooden casks, i.e., the seasoning time, have a particular effect on the wood from the casks.

Fino Sherry is a dry fortified wine (15% Alcohol by Volume (ABV)), obtained by biological ageing under the action of flor velum yeasts, and it is characterized by its pale-yellow colour, slightly acid flavour, and sharp aromas with hints of almonds (Valcárcel-Muñoz et al. 2022b) also a dry fortified wine (18–20% ABV) obtained by oxidative ageing. It develops an amber-mahogany colour, and its aroma evolves differently from that of Fino Sherry wine (Zea et al. 2001, 2013; Durán-Guerrero et al. 2021; García-Moreno et al. 2021; Valcárcel-Muñoz et al.

2022a) fortified wine (15–17% ABV) obtained through an oxidative ageing process. It is very rich in sugars (more than 400 g/L), as a consequence of the raising process applied to the grapes. Pedro Ximénez Sherry wine is only partially fermented, it is immediately fortified in order to preserve its original sweetness and to ensure ageing in an oxidative environment. This enhances its complexity, while an ebony colour and a dense appearance are acquired (Ruiz-Bejarano et al. 2016; Herrera et al. 2020; Durán-Guerrero et al. 2021; Valcárcel-Muñoz et al. 2023).

Oak wood consists of 90% cellulose, hemicellulose, and lignin. Therefore, the compounds derived from it, such as furfural or its derivatives (whose origin is the hemicellulose) (Sarni et al. 1990; Le Floch et al. 2015) and guaiacyl-type aldehydes (vanillin and coniferylaldehyde) and syringyl-type aldehydes (syringaldehyde and sinapaldehyde), and cinnamic and benzoic acids (Mosedale and Puech 1998; Canas 2017) (from the lignin), make up the most significant extractable fractions of this wood. The remaining 10% of the wood composition consists of tannins, other phenolic compounds (polyphenols or simpler phenols), short-chain carboxylic acids, fatty acids, alcohols, or inorganic substances (Mosedale and Puech 1998).

Therefore, wood casks are containers that interact with the product during its ageing. The concentration and availability of certain compounds from wood depend on certain factors intrinsic to the wood itself, such as its geographical origin or its botanical species (Jordao et al. 2005; Prida and Puech 2006; Martínez-Gil et al. 2018), wood toast (Soares et al. 2012; Canas 2017; Martínez-Gil et al. 2018), and barrel volume (Pérez-Prieto et al. 2002), as well as on those related to the extraction process. These are inherent to the particular ageing beverage and its alcohol strength and/or to the specific climatic conditions—including temperature and humidity—, under which the ageing process takes place.

Casks yield a large number of compounds into the beverages that they may contain (wines, vinegars or distillates, among others), and these compounds are involved in numerous hydrolysis, oxidation, esterification, ethanolysis, polymerization, polycondensation and Maillard reactions (Nishimura and Matsuyama 1989; Mosedale and Puech 1998). Other physical phenomenon that takes place during the ageing of beverages is the evaporation or transpiration of water through the pores of the wood and that has an impact on the final product.

Likewise, in the case of seasoned woods, not only the woods' own compounds contribute to these beverages, but also other compounds that come from the wines used for their seasoning. Thus, the wood of the casks acts as a transfer vector between the two liquids. In other words, the Sherry wines used to fill these barrels give up some of their constituents to wood. These constituents retained in the wood pores become available to the beverages that will later be aged inside the casks. This process enriches the beverages aged in *Sherry Casks*® with a unique noble and distinctive character. Figure 1 includes several digital microscope images of the surface of cask staves that have been previously seasoned with Sherry wines. The crystals that can be observed in these photographs are explained by the precipitation of tartaric acid and/or other compounds that are present in the wine and which have penetrated the wood pores during the seasoning process.

Given the current growing trend to use *Sherry Casks*® for the ageing of certain products with the purpose of providing them with a superior quality as well as

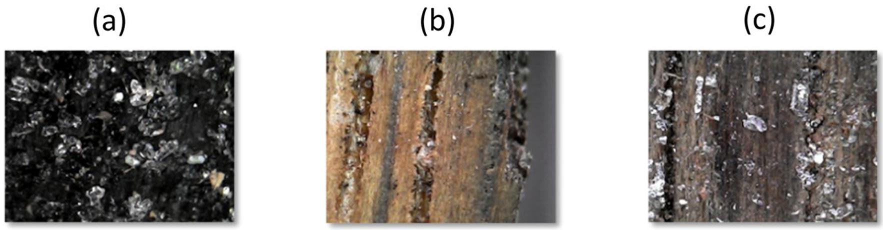

Fig. 1 Digital microscope images of the surface in contact with the wine of the *Sherry Casks*® staves with tartaric acid precipitates, among others. From left to right is the surface of a stave with **a** heavy toast, **b** light toast, and **c** medium toast

distinctive flavors and aromas, and the lack of studies on the impact that the seasoning process has on the wood of the barrels, we have considered that it would be of interest to investigate the volatile composition of the wood of the *Sherry Casks*® after the seasoning process has been completed. The aim of this study is to determine how this seasoning process affects the compounds that are originally present in the wood and which ones of the wines' own compounds — which are not present in the unseasoned wood—, remain attached to the wood so that they become susceptible of being transferred into the products that are later on aged in these casks.

For this purpose, we have studied the wood of *Sherry Casks*® that have been seasoned with the most commonly used Sherry wines, such as Fino, Oloroso, and Pedro Ximénez. The research has been carried out on woods that had been seasoned with these wines for at least 12 months, which is the minimum time required by the applicable Technical Specifications to be granted a *Sherry Casks*® certification (Regulatory Council 2021). We have also used wood from casks that had been seasoned with these wines for 5 years or more, since there are certain higher categories of products that require the use of casks that have been seasoned for a longer period of time. In this way, we have evaluated casks seasoned for the shortest time required and also others seasoned for a longer period of time. Direct Thermal Desorption–Gas Chromatography–Mass Spectrometry (DTD–GC–MS) has been applied to wood grindings to determine the volatile composition of the different woods (Guerrero-Chanivet et al. 2020).

Materials and methods

Samples and preparation (woods and wines)

All the wood fragments studied were part of staves from medium toasted American oak (*Quercus alba*) casks that had been filled with one of three types of Sherry wines: Fino, Oloroso, or Pedro Ximénez.

The 12-month seasoned casks used for the study were new medium-toasted American oak (*Quercus alba*) casks with a capacity of 500 L (Tonelería Huberto Domecq, Jerez de la Frontera, Spain). The toasting of the wood was carried out according to the traditional artisan process used by the supplying company. The vessels filled for 5 years with Fino, Oloroso and Pedro Ximénez Sherry wines belonged to the industrial *solerajes* of Bodegas Fundador S.L.U., all of which were made of medium toasted American oak (*Quercus alba*). One unseasoned barrel, supplied by the same cooperage was used as the reference in the study. All of the Sherry wines used to season the casks complied with the technical specifications set out in the applicable regulations for these wines (Council of Agriculture Fisheries and Rural Development 2022) and were supplied by Bodegas Fundador, S.L.U., as well as the cellar where all the seasoning process was carried out.

After the stipulated ageing time (12 or 60 months), all the wines contained in the casks were removed and the casks were let to drain upside down for 72 h in order to eliminate any liquid remains from them. Then, a bottom stave was removed from each barrel. A fragment from the central area of each stave was cut out in 3 layers. The innermost layer, i.e., the one in contact with the wine (6.3–8.5 mm long × 3.1–3.6 mm wide × 0.5–0.75 mm thick) was ground to 0.25 µm grain size powder by means of an ultracentrifugal mill ZM 200 (Retsch GmbH, Haan, Germany) before analysis.

DTD–GC–MS analysis of the volatile compounds in the wood grindings

The analysis of the oak wood volatile compounds was carried out by DTD–GC–MS following the method proposed by Guerrero-Chanivet et al. (2020). For this purpose, 10 mg of wood grindings were placed in desorption tubes together with 5 µL of IS (Internal Standard, 303 mg L^{-1} solution of 4-methyl-2-pentanol) (Sigma-Aldrich, Steinheim, Germany) in 40% ethanol–water ethanol solution (Sigma-Aldrich, Saint Louis, MO, USA), capped at both ends with silanized glass wool. The desorption tubes were heated up to 250 °C for 7 min. The volatile compounds were desorbed in a helium stream (Air Liquide España, S.A., Madrid, Spain) and collected in a cold trap (−15 °C). The desorption was carried out at 250 °C for 6 min and the volatile compounds were transferred (1:10 split) into the chromatographic column through a line heated at 225 °C. The GC oven gradient was as follows: held at 35 °C for 10 min and then raised at 5 °C per minute up to 100 °C. It was then raised to 210 °C at 3 °C per minute 1 and held for 40 min. The analyses were carried out by means of a Shimadzu GCMS-TQ8040 gas chromatographer with mass detection (Shimadzu, Kyoto, Japan) equipped with a DB-Wax 60 m × 0.25 mm internal diameter capillary column (J&W Scientific, Folsom, CA, USA), with a 0.25 µm coating. The samples were analyzed in duplicate. The compounds were identified by analogy with the mass spectra kept by LabSolutions GCMSsolution (Shimadzu, Kyoto, Japan). The relative area of each compound was obtained by measuring the area of the chromatographic signal produced by the fragment with the highest mass (base peak) with respect to that of IS, 4-methyl-2-pentanol. The results were expressed as relative areas.

Statistical analysis

Statgraphics 19 software package (Statgraphics Technologies, Inc., The Plains, VA, USA) was used for ANOVA and Fisher's least significant difference test. The other statistical parameters were calculated using Microsoft Excel 2016 (Microsoft Corp., Redmond, WA, USA).

Results and discussion

Figure 2a–c shows the chromatographic profiles of the oak wood seasoned for 12 months and 60 months, and the reference of the unseasoned wood, for each type of seasoning (Fino, Oloroso and Pedro Ximénez Sherry wines). As can be seen in this figure, each wood presents a different profile, depending on the Sherry wine it contained. In addition, certain differences in the intensity of the peaks can also be observed depending on the length of time that the Sherry wine remained in the casks. The most relevant compounds determined in this analysis for each one of the wood samples studied are shown in Tables 1, 2 and 3. All of their values have been expressed as units of relative area with respect to the internal standard 4-methyl-2-pentanol. In general, it can be said that some of the compounds that are naturally present in the wood, either disappeared or decreased their concentration as they were extracted by the seasoning wines. On the other hand, some typical wine compounds (which may or may not be part of the wood composition) increased their concentrations or appeared in the wood during the seasoning process, either as a consequence of the penetration of the wine into the wood pores or because of the precipitation of certain substances. These compounds that were contributed by the wines to the wood differed according to the seasoning Sherry wine used.

Compounds that disappeared from the wood during seasoning

In all the samples studied, i.e. those seasoned for 12 or 60 months using different Sherry wines and the unseasoned one, the characteristic compounds of the oak wood were detected. This is in agreement with the results published in previous studies (Guerrero-Chanivet et al. 2020), whose relative area values are shown in Table 1. Only three compounds were detected in the unseasoned wood that were not detected in the others: levulinic acid, vanillic acid and vanillyl methyl ketone. These three compounds, which are part of the composition of the wood, are probably extracted from the wood by the seasoning wines, which results in their decreasing concentration to the extent of not being detectable in the seasoned woods.

The degradation of the furan-derivatives, that are formed from wood pentoses and hexoses, yields levulinic acid (Jönsson et al. 2013; Geffert et al. 2019). Vanillic acid and vanillyl methyl ketone are two compounds derived from vanillin, which is a guaiacyl-type aldehyde that originates from the degradation of wood lignin and is closely related to the heat treatment that these casks are subjected to (Mosedale and

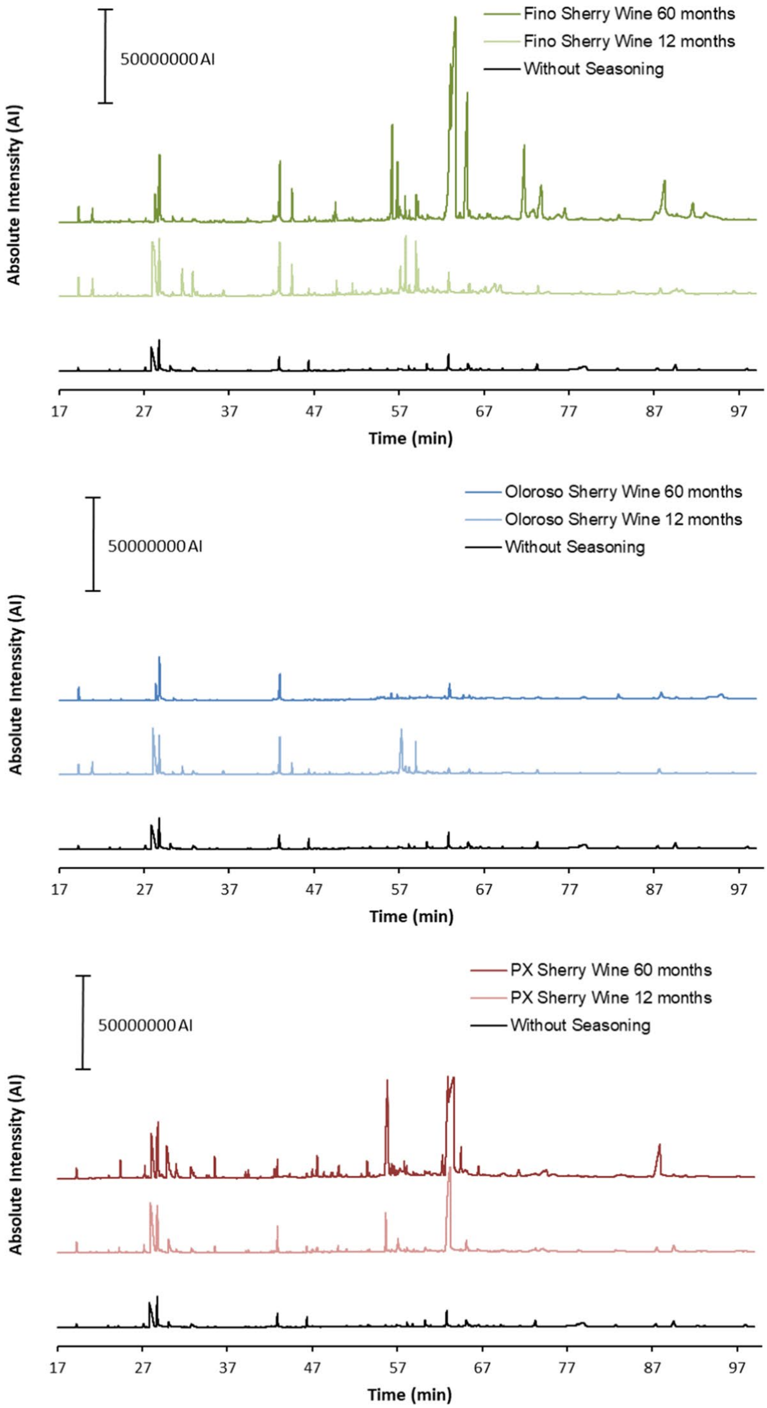

Fig. 2 Chromatographic profiles of wood seasoned with Sherry wines for 12 months and 60 months, compared to unseasoned wood of **a** Fino Sherry Wine, **b** Oloroso Sherry Wine and **c** Pedro Ximénez (PX) Sherry Wine

Table 1 Relative area values of volatile compounds in non-seasoned *Quercus alba* medium toast stave determined by DTD–GC–MS that disappeared from the wood during the seasoning

RT (min)	Compound	Relative area value
57.337	Levulinic acid	0.278 ± 0.025
59.258	Vanillic acid	0.183 ± 0.011
69.178	Vanillyl methyl ketone	0.722 ± 0.014

Puech 1998; Canas 2017). In fact, several studies claim that vanillic acid is transferred from the wood into the ageing beverage (Valcárcel-Muñoz et al. 2022a, b), which corroborates this hypothesis.

Compounds that change their concentration levels during the seasoning

Compounds that reduce their concentration levels during the seasoning

When focusing on other types of compounds in the unseasoned wood with respect to the seasoned woods (Guerrero-Chanivet et al. 2020), it can be observed that such compounds are found in greater proportions in the former ones. This is due to the fact that, during the seasoning process, the wine is not only in contact with the surface of the staves, but it actually penetrates the wood pores and extracts certain compounds from it, thus decreasing the concentration of these compounds in the seasoned woods (García-Moreno et al. 2021). Table 2 shows the relative area values of volatile compounds that increase or decrease during the seasoning process.

Among the compounds detected, acetic acid is one of those with the greatest relative area in all the woods studied. This compound is obtained through the degradation of hemicellulose (Ruiz et al. 2017) and is observed to decrease after the seasoning. This decrease was more pronounced in the woods that had been seasoned for 60 months (a longer time in contact with the wine, a greater extraction of components) than in the woods that had been seasoned for just 12 months. This compound reduction was less noticeable in the woods seasoned using Pedro Ximénez Sherry wines. This is explained by the fact that this sweet Sherry wine has a greater density and viscosity because of its high sugar content. Thereby, its hygroscopicity hinders its penetration into the wood pores and, consequently, it achieves a poorer extraction. Consequently, the extraction of polyphenols from the pores of the wood is significantly higher when dry wines, rather than sweet wines, are used for seasoning purposes (González-Gordon 2005; Barquín 2019; Valcárcel-Muñoz et al. 2023).

Furfural and its derivatives, which come from the degradation of hemicellulose, are also noteworthy. Furfural, in particular, presented a large relative area value with respect to the rest of the compounds. These compounds decreased in the woods that had been seasoned with Fino or Oloroso, both for 12 and the 60-month-seasoned casks. However, it is worth noting the particular case of the wood seasoned with Pedro Ximénez, where the reduction of furfural was not so marked and even some of its derivatives increased their concentration over the seasoning period. Moreover, the longer the seasoning time (60 months), the greater the presence of these compounds in the final wood. This would be the case of 5-methylfurfural, furfuryl

Table 2 Relative area values of volatile compounds that evolve during the seasoning determined by DTD–GC–MS

RT (min)	Compound	Without seasoning	Fino Sherry Wine		Oloroso Sherry Wine		PX Sherry Wine	
			12 months	60 months	12 months	60 months	12 months	60 months
Compounds that disappeared from the wood during the seasoning								
23.577	Acetoin	0.033 ± 0.001^{c}	0.095 ± 0.004^{e}	0.022 ± 0.006^{b}	0.018 ± 0.004^{b}	0.005 ± 0.001^{a}	0.043 ± 0.005^{d}	n.d
24.065	Acetol	0.320 ± 0.008^{b}	0.046 ± 0.001^{a}	0.049 ± 0.000^{a}	0.051 ± 0.001^{a}	0.052 ± 0.001^{a}	0.270 ± 0.056^{ab}	0.965 ± 0.286^{c}
27.112	Methyl 2-propanoate	0.635 ± 0.079^{b}	0.066 ± 0.006^{a}	0.152 ± 0.033^{a}	0.100 ± 0.010^{a}	0.051 ± 0.004^{a}	0.491 ± 0.143^{b}	1.035 ± 0.178^{c}
27.952	Acetic acid	22.567 ± 1.051^{e}	8.621 ± 0.900^{b}	1.738 ± 0.215^{a}	10.520 ± 0.636^{c}	1.046 ± 0.026^{a}	13.308 ± 0.343^{d}	8.168 ± 0.747^{b}
28.773	Furfural	10.731 ± 0.314^{d}	3.246 ± 0.384^{a}	4.682 ± 0.296^{b}	3.353 ± 0.037^{a}	3.401 ± 0.193^{a}	5.297 ± 0.287^{b}	9.755 ± 0.859^{c}
30.103	Formic acid	2.372 ± 0.186^{b}	0.299 ± 0.008^{a}	0.469 ± 0.081^{a}	0.321 ± 0.006^{a}	0.246 ± 0.020^{a}	2.074 ± 0.054^{b}	8.450 ± 0.534^{c}
31.228	Propanoic acid	0.117 ± 0.008^{c}	0.027 ± 0.002^{b}	0.026 ± 0.004^{b}	0.016 ± 0.001^{a}	0.015 ± 0.001^{a}	n.d	n.d
32.892	5-Methylfurfural	1.007 ± 0.109^{c}	0.167 ± 0.030^{a}	0.109 ± 0.021^{a}	0.061 ± 0.006^{a}	0.093 ± 0.056^{a}	0.636 ± 0.051^{b}	1.802 ± 0.139^{d}
34.640	2-Propenoic acid	0.116 ± 0.009^{d}	0.031 ± 0.003^{a}	0.078 ± 0.006^{c}	0.041 ± 0.005^{ab}	0.021 ± 0.001^{a}	0.059 ± 0.001^{bc}	0.237 ± 0.019^{e}
35.530	Furfuryl alcohol	0.340 ± 0.002^{b}	0.035 ± 0.022^{a}	0.061 ± 0.008^{a}	0.059 ± 0.003^{a}	0.042 ± 0.008^{a}	0.593 ± 0.031^{c}	2.041 ± 0.139^{d}
40.923	Heptanal	0.057 ± 0.006^{c}	n.d	n.d	0.014 ± 0.003^{a}	0.010 ± 0.001^{a}	0.034 ± 0.003^{b}	0.117 ± 0.002^{d}
44.065	*trans*-Whiskey lactone	0.131 ± 0.001^{d}	0.089 ± 0.007^{c}	0.039 ± 0.016^{a}	0.062 ± 0.003^{b}	n.d	0.027 ± 0.006^{a}	n.d
46.355	*cis*-Whiskey lactone	2.565 ± 0.121^{c}	0.279 ± 0.025^{a}	0.287 ± 0.039^{a}	0.455 ± 0.025^{b}	n.d	0.525 ± 0.044^{b}	n.d
47.595	Methyl 2-furoate	0.140 ± 0.003^{a}	n.d	n.d	n.d	n.d	0.299 ± 0.014^{b}	1.432 ± 0.120^{c}
52.648	Eugenol	0.194 ± 0.011^{b}	n.d	n.d	0.144 ± 0.014^{a}	n.d	n.d	n.d
55.588	Pyranone	0.355 ± 0.013^{a}	0.147 ± 0.044^{a}	n.d	0.096 ± 0.009^{a}	0.024 ± 0.002^{a}	2.366 ± 0.395^{b}	16.069 ± 0.427^{c}
55.725	Syringol	0.260 ± 0.022^{b}	0.024 ± 0.001^{a}	n.d	0.029 ± 0.002^{a}	n.d	n.d	n.d
58.232	*Trans*-Isoeugenol	0.899 ± 0.102	0.256 ± 0.051	2.492 ± 2.867	n.d	n.d	n.d	n.d
60.352	2-Furoic acid	2.224 ± 0.065^{c}	n.d	n.d	n.d	n.d	0.645 ± 0.016^{b}	0.503 ± 0.015^{a}
65.215	Vanillin	1.492 ± 0.180^{d}	0.780 ± 0.078^{c}	n.d	0.569 ± 0.047^{b}	0.251 ± 0.023^{a}	0.763 ± 0.004^{c}	n.d
73.283	Methoxyeugenol	2.631 ± 0.164^{ab}	0.484 ± 0.069^{a}	3.504 ± 3.170^{b}	0.517 ± 0.019^{a}	0.192 ± 0.016^{a}	0.368 ± 0.159^{a}	n.d
78.215	Butyrovanillone	1.270 ± 0.072^{b}	n.d	n.d	0.140 ± 0.020^{a}	0.192 ± 0.062^{a}	n.d	n.d
89.640	Syringaldehyde	4.289 ± 0.138^{d}	0.429 ± 0.064^{b}	n.d	0.214 ± 0.059^{a}	0.320 ± 0.043^{ab}	1.547 ± 0.165^{c}	n.d

Table 2 (continued)

RT (min)	Compound	Without seasoning	Fino Sherry Wine		Oloroso Sherry Wine		PX Sherry Wine	
			12 months	60 months	12 months	60 months	12 months	60 months
Compounds that increase their concentration levels during the seasoning								
39.198	2(5H)-Furanone	0.063 ± 0.001^a	n.d	0.213 ± 0.084^b	0.053 ± 0.002^a	0.014 ± 0.001^a	0.185 ± 0.004^b	0.703 ± 0.053^c
63.110	5-HMF	6.261 ± 0.547^b	1.272 ± 0.211^a	2.196 ± 1.764^a	0.703 ± 0.018^a	1.569 ± 0.183^a	24.821 ± 2.199^c	67.011 ± 1.717^d
87.437	Palmitic acid	1.936 ± 0.189^a	0.923 ± 0.109^a	8.081 ± 3.015^b	1.266 ± 0.114^a	1.190 ± 0.046^a	1.228 ± 0.465^a	11.407 ± 1.106^c

Mean values ± standard deviations are shown; ANOVA: different letters indicate significant differences ($p < 0.05$). *PX* Pedro Ximénez; *n.d.* not detected

Table 3 Relative area values of volatile compounds that appeared during the seasoning determined by DTD–GC–MS

RT (min)	Compound	Fino Sherry Wine		Oloroso Sherry Wine		PX Sherry Wine	
		12 months	60 months	12 months	60 months	12 months	60 months
20.72	2-Methyl-1-butanol	0.133 ± 0.019^{bc}	0.121 ± 0.012^{b}	0.175 ± 0.014^{c}	0.008 ± 0.001^{a}	0.025 ± 0.000^{a}	0.083 ± 0.047^{b}
20.83	3-Methyl-1-butanol	0.781 ± 0.092^{bc}	0.670 ± 0.061^{b}	0.917 ± 0.106^{c}	0.054 ± 0.001^{a}	0.088 ± 0.005^{a}	0.081 ± 0.009^{a}
31.08	Methyl-malonic acid	n.d	n.d	n.d	n.d	0.278 ± 0.055^{a}	0.559 ± 0.001^{b}
31.52	2,3-Butanediol	1.688 ± 0.361^{c}	0.106 ± 0.043^{a}	0.855 ± 0.071^{b}	n.d	n.d	n.d
33.28	Lactic acid	0.176 ± 0.013^{b}	n.d	0.067 ± 0.007^{a}	n.d	n.d	0.208 ± 0.074^{b}
34.442	Hexanoic acid	0.034 ± 0.003^{d}	0.022 ± 0.001^{b}	0.027 ± 0.000^{c}	0.012 ± 0.003^{a}	n.d	n.d
34.923	4-Hydroxybutanoic acid	0.101 ± 0.007^{c}	0.081 ± 0.008^{c}	0.041 ± 0.004^{b}	0.008 ± 0.002^{a}	0.036 ± 0.004^{ab}	0.222 ± 0.029^{d}
35.337	Ethyl Decanoate	0.017 ± 0.001^{b}	0.090 ± 0.001^{d}	0.008 ± 0.003^{a}	n.d	n.d	0.059 ± 0.007^{c}
36.343	Diethyl succinate	0.456 ± 0.024^{c}	0.221 ± 0.010^{b}	0.502 ± 0.064^{c}	n.d	n.d	0.055 ± 0.029^{a}
44.412	Phenylethyl Alcohol	1.612 ± 0.129^{d}	1.949 ± 0.021^{e}	0.994 ± 0.100^{c}	0.088 ± 0.001^{a}	0.059 ± 0.000^{a}	0.286 ± 0.022^{b}
48.837	Diethyl dl-malate	n.d	n.d	0.160 ± 0.012	n.d	n.d	n.d
49.231	Octanoic acid	0.114 ± 0.006^{b}	0.205 ± 0.000^{c}	0.102 ± 0.016^{b}	0.022 ± 0.000^{a}	n.d	0.397 ± 0.026^{d}
49.528	Ethyl myristate	n.d	1.085 ± 0.005	n.d	n.d	n.d	n.d
55.883	n-Decanoic acid	0.037 ± 0.001^{b}	n.d	0.020 ± 0.000^{a}	n.d	n.d	n.d
56.095	Ethyl palmitate	0.088 ± 0.001^{a}	7.076 ± 0.889^{c}	n.d	0.360 ± 0.048^{a}	0.086 ± 0.005^{a}	1.901 ± 0.252^{b}
56.332	3,5-Dihydroxy-2-methyl-4-pyrone	n.d	n.d	n.d	n.d	0.165 ± 0.000^{a}	0.883 ± 0.054^{b}
56.778	9-Hexadecenoic acid, ethyl ester	n.d	4.637 ± 0.956	n.d	n.d	n.d	n.d
58.211	Diethyl L-tartrate	n.d	n.d	0.310 ± 0.028^{b}	0.114 ± 0.006^{a}	n.d	n.d
59.013	Ethyl hydrogen succinate	1.647 ± 0.093^{c}	0.917 ± 0.113^{b}	1.592 ± 0.243^{c}	0.025 ± 0.003^{a}	0.091 ± 0.017^{a}	n.d
62.803	Ethyl stearate	n.d	n.d	n.d	n.d	0.093 ± 0.006^{a}	2.416 ± 0.091^{b}
63.53	Ethyl 9-octadecenoate	n.d	47.008 ± 2.870	n.d	n.d	n.d	n.d
80.738	3-hydroxylauric acid	n.d	0.332 ± 0.033	n.d	n.d	n.d	n.d

Mean values ± standard deviations are shown; ANOVA: different letters indicate significant differences ($p < 0.05$). *PX* Pedro Ximénez; *n.d.* not detected alcohol and methyl 2-furoate. This is explained by the fact that the Pedro Ximénez Sherry wine that penetrates the pores of the wood and the wine that remains adhered to the surface of the staves is much richer in phenolic content than other Sherry wines because of the high concentration of polyphenols and furanic aldehydes reached by the raisins used to make Pedro Ximénez. These raisins are obtained through a drying process that consists of their direct sunlight exposure (Chaves et al. 2007; Ruiz et al. 2010; Herrera et al. 2020).

Regarding formic acid, a decrease was observed in all the seasoned woods with respect to the unseasoned wood, except in the case of those casks seasoned with Pedro Ximénez. Thus, in the 12-month seasoned wood, we could say that it remained seemingly unaltered; however, the wood that had been seasoned for 60 months revealed an increment of this compound. Formic acid can be generated by Maillard reactions (van Boekel 2006). Since Pedro Ximénez Sherry wines contain large amounts of sugars, formic acid formation may be more favored than in the rest of the wines under study, which is consistent with the observed trend.

Cis- and *trans*-whiskey lactones are two very common compounds in oak wood, being more abundant in *Quercus alba*. A decrease of these compounds was observed in the three seasoned woods, and they were no longer detectable in the woods treated with Oloroso or Pedro Ximénez for the longest period.

Some syringyl-type aldehydes such as syringaldehyde, which also come from the degradation of lignin, follow the aforementioned trend, and were even undetectable in the long-term seasoned woods that used Fino or Oloroso.

The levels of pyranone found in the woods seasoned with Pedro Ximénez and particularly in the one seasoned for 60 months, were also noteworthy when compared against the rest of the woods analyzed. Since this compound is also related to the Maillard reactions that may take place in the wines (Mottram 2007; Gomes Pereira 2011) and given the large quantities of sugars—which are compounds involved in the Maillard reactions— that are found in Pedro Ximénez sweet Sherry wines, the amount of pyranone detected in these woods was much higher than that found in the rest of the woods (Mottram 2007; Gomes Pereira 2011).

In general and, taking into account that wood is a very heterogeneous material, a decrease in the concentration of most of the compounds that are part of the composition of wood was observed—with the already mentioned exceptions—, as a result of their extraction by the wines used to season the casks. This fact is more pronounced in those woods subjected to 60-month seasoning compared against those that were seasoned for just 12 months. Thus, the percentage of compounds that were present in unseasoned and then undetected after the seasoning, was higher in the case of long-seasoned woods. In addition to the particular case of the proportion of furans and derivatives in the wood seasoned with Pedro Ximénez, a greater quantity of other compounds from the wood was generally detected in these woods in comparison with those seasoned with other Sherry wines for the same amount of time. This may be due to the fact that Pedro Ximénez is a much denser wine because of its high sugar content, which may hinder its penetration into the wood pores and makes compound extractions more difficult (González-Gordon 2005; Barquín 2019; Valcárcel-Muñoz et al. 2023).

Compounds that increase their concentration levels during the seasoning

In this section we should highlight those compounds that are part of the composition of the wood but that, in general, as a result of the wood being seasoned with the Sherry wines, increments in the concentration levels of these compounds was observed. The relative area values of these compounds are also shown in Table 2. The relevant compounds are 2(5H)-Furanone, 5-HMF and palmitic acid.

2(5H)-Furanone is part of the compound 3-Hydroxy-4,5-dimethyl-2(5H)-furanone, commonly known as sotolone. This lactone is a very odorous compound and is responsible for spiciness and curry flavors in wines and is formed by the oxidation of certain compounds resulting from the yeast cells' metabolism, or lysis (Roldán Gómez 2008). It is actually related to yeast metabolism and can be found in Fino Sherry wines, which are biologically aged under flor velum yeast (Martin et al. 1992; Collin et al. 2012). This compound is also associated with the degradation of sugars (Gaspar et al. 2019), which explains why it has been detected in other studies on Pedro Ximénez wines in higher amounts than in other Sherry wines, such as Fino (Campo et al. 2008). This fact is consistent with the fact that 2(5H)-Furanone increased its concentration more markedly in the wood aged for 60 months with Pedro Ximénez, followed by the same wood aged for just 12 months, and by the wood aged for longer time with Fino Sherry wine.

Note that 5-HMF is part of the natural composition of wood and that is closely related to the degradation of the hemicellulose that takes place as a result of the thermal treatment applied to the wood used to manufacture the casks. 5-HMF decreased in all of the 12 and 60-month seasoned casks that were seasoned with Fino or Oloroso but increased considerably in those that had been aged with Pedro Ximénez, being this increment much more noticeable in the casks that had been seasoned for 60 months. As already mentioned, this can be explained by the large amounts of polyphenol (Valcárcel-Muñoz et al. 2022b) and furanic aldehydes deposited on the wooden staves and into its pores by the Pedro Ximénez Sherry wines as a result of their production with raisined grapes directly exposed to sunlight (Chaves et al. 2007; Ruiz et al. 2010, 2014; Herrera et al. 2020).

A considerable increase of palmitic acid contents was observed in two of the woods studied as follows: the wood treated for 60 months with Pedro Ximénez and — even if slightly less — the wood treated for 60 months with Fino Sherry wine. In the particular case of Pedro Ximénez, the incremental concentration that was detected in the wood over its seasoning (Agudelo-Romero et al. 2013) was a result of the adhesion and penetration of this wine into the wood. On the other hand, the greater concentration of this compound in the wood treated with Fino Sherry wine for the longest period is attributable to the composition of the lipids from Sherry wine lees (Agudelo-Romero et al. 2013), as it is aged under flor velum yeasts (Valcárcel-Muñoz et al. 2022b). In this case, a longer seasoning process allows for the dying yeasts to deposit and stick to the staves, thus increasing the concentration of palmitic acid in the wood. On the other hand, a 12-month seasoning time did not seem to be long enough to allow for this process to take place, or at least not to a significant extent.

Compounds that appear during seasoning

A series of compounds were only present in the wine-seasoned wood and were not detected in the wood that did not previously contain any type of wine. The relative area values of all these compounds are shown in Table 3 for each case studied. These compounds are naturally found in the wines and, as a consequence of the seasoning process, they are retained in the pores of the wood or attached to its surface. Among these compounds we find the following: 2-methyl-1-butanol; 3-methyl-1-butanol; ethyl lactate; methyl-malonic acid; 2,3-butanediol; lactic acid; hexanoic acid; 4-hydroxybutanoic acid; ethyl decanoate; diethyl succinate; phenylethyl alcohol; diethyl dl-malate; octanoic acid; n-decanoic acid; ethyl palmitate; 3,5-Dihydroxy-2-methyl-4-pyrone; diethyl L-tartrate; ethyl hydrogen succinate and ethyl stearate. Most of the compounds above-mentioned are isoamyl alcohols, organic acids or esters present in the Sherry wines used for the seasoning.

Isoamyl alcohols (2-methyl-1-butanol and 3-methyl-1-butanol) are originated by different metabolic pathways of decarboxylation/deamination of amino acids (Swiegers et al. 2005; Pozo-Bayón and Moreno-Arribas 2011). In the case of Pedro Ximénez Sherry wines, the alcoholic fermentation was minimal and, therefore, the concentration of these compounds was lower than in the oxidative aged Sherry wines (Martínez de la Ossa et al. 1987; Valcárcel-Muñoz et al. 2022a, b) where must is completely fermented. Therefore, these two higher alcohols were found at greater concentrations in the Fino and Oloroso Sherry wines seasoned woods rather than in the Pedro Ximénez-seasoned ones.

2,3-butanediol is an aromatic compound formed from acetaldehyde (Avdanina and Zghun 2022). Acetaldehyde is associated with the metabolism of flor yeasts (Moreno et al. 2005), and therefore reaches higher concentrations in the Sherry wines that undergo biological ageing (Valcárcel-Muñoz et al. 2022b). This explains why this compound's highest relative area value (1.688) was found in the wood aged with Fino Sherry wine for 12 months. With respect to ethyl palmitate, the large amounts found in wood aged with Fino Sherry wine for 60 months are noteworthy. As previously mentioned, palmitic acid is part of the composition of the lipids from the lees used to age Fino Sherry wines (Agudelo-Romero et al. 2013) and, in the presence of ethanol, it is esterified into the corresponding ester.

In addition to the aforementioned compounds, which are typical of the seasoning process, a series of compounds—that were not found in the other types of wood—were detected in the wood seasoned for 60 months with Fino Sherry wine: ethyl myristate (relative area: 1.085); ethyl 9-hexadecenoate (relative area: 4.637); ethyl 9-octadecenoate (relative area: 47.008) and 3-hydroxylauric acid (relative area 0.332). Fino Sherry wine is aged under biological ageing in the presence of flor yeasts (Valcárcel-Muñoz et al. 2022b) and all these compounds are related to the metabolism of flor yeasts (Moreno et al. 2005) and to their composition (Agudelo-Romero et al. 2013). Among those mentioned, ethyl 9-octadecenoate stands out with the highest relative area value of all the compounds identified and appears at 63.53 min. The presence of oleic acid in the membranes of Saccharomyces cerevisiae (flor velum yeasts) has been demonstrated in the literature (Daum et al. 1998; You et al. 2003; Duan et al. 2015). Once the lysis of the yeasts takes place, this fatty acid is transferred into the medium and can be esterified by the ethanol that is found in it, giving rise to this ethyl ester. These compounds are typical of the yeasts' cell walls under which these wines are aged. As cells die, these compounds are gradually deposited onto the bottom of the casks, where they remain attached to the staves. This hypothesis was experimentally corroborated by doping unseasoned wood with flor yeasts.

These compounds would not normally be present in wood that has not been aged for a considerable time with Sherry wine. This makes these barrels that contain the memory of their seasoning wines a very special product as they confer unique organoleptic characteristics to the distillates that are subsequently aged inside them.

Conclusion

By applying a novel DTD–GC–MS method, we have been able to characterize the volatile composition of barrel staves seasoned using Fino, Oloroso or Pedro Ximénez Sherry wines. Different profiles of volatile compounds have been determined, depending on whether the wood had been seasoned or not and depending on the type of Sherry wine used for the seasoning process. In addition, certain differences have been revealed in accordance with the length of time that the seasoning wine remained inside the casks.

As a general tendency, those compounds that are naturally present in these woods reduced their concentrations as a result of the seasoning process, since the wines, while inside the cask, extracted part of these compounds from the wood. It has been observed that, the longer the time the wine remains inside the cask, the lower the number of compounds exclusively from the wood are detected.

On the other hand, those compounds that are naturally present in the wood and in the wines, have seen their concentration increased as the seasoning wines penetrated the wood pores and enriched the containers with these compounds.

Note that numerous compounds typical of Sherry wines have been found in seasoned wood, including isoamyl alcohols (2-methyl-1-butanol and 3-methyl-1-butanol), organic acids and esters and/or compounds derived from them. These compounds owe their presence in the wood exclusively to the seasoning process, which makes *Sherry Casks*® highly valuable pieces of equipment. Furthermore, the compounds that have been found in seasoned wood, were consistent with the profile of the wine used for their seasoning. Thus, the typical compounds that are present in biological ageing processes and/or flor velum yeasts, were found in Fino-seasoned woods, while the compounds that are typical of oxidative ageing were found in Oloroso and Pedro Ximénez-seasoned wood, and in the latter, these compounds owed their presence to the large quantities of sugars contained in this wine.

To sum up, it has been demonstrated that, during their ageing, Sherry wines can contribute valuable compounds to the wood of the casks containing them. These compounds, which are retained by the wood are later on transferred into the distillates, as they undergo their own ageing process. Thus, conferring unique quality traits on these spirits and making them different from any other beverage aged in unseasoned wood.

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Declarations

Competing interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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