



Volatile profile of Madeira wines submitted to traditional accelerated ageing



Vanda Pereira^{a,b,*}, Juan Cacho^c, José C. Marques^{a,b}

^aCentre of Exact Sciences and Engineering, University of Madeira, Campus da Penteada, 9000-390 Funchal, Portugal

^bInstitute of Nanostructures, Nanomodelling and Nanofabrication (I3N), University of Aveiro, Aveiro, Portugal

^cDepartment of Analytical Chemistry, Faculty of Sciences, University of Zaragoza, 50009 Zaragoza, Spain

ARTICLE INFO

Article history:

Received 17 December 2013

Received in revised form 8 April 2014

Accepted 9 April 2014

Available online 18 April 2014

Keywords:

Fortified wines

Accelerated ageing

Heating

Volatiles

ABSTRACT

The evolution of monovarietal fortified Madeira wines forced-aged by traditional thermal processing (*estufagem*) were studied in terms of volatiles. SPE extracts were analysed by GC–MS before and after heating at 45 °C for 3 months (standard) and at 70 °C for 1 month (overheating). One hundred and ninety volatile compounds were identified, 53 of which were only encountered in baked wines. Most chemical families increased after standard heating, especially furans and esters, up to 61 and 3-fold, respectively. On the contrary, alcohols, acetates and fatty acids decreased after heating. Varietal aromas, such as Malvasia's monoterpenic alcohols were not detected after baking. The accelerated ageing favoured the development of some volatiles previously reported as typical aromas of finest Madeira wines, particularly phenylacetaldehyde, β -damascenone and 5-ethoxymethylfurfural. Additionally, ethyl butyrate, ethyl 2-methylbutyrate, ethyl caproate, ethyl isovalerate, guaiacol, 5-hydroxymethylfurfural and γ -decalactone were also found as potential contributors to the global aroma of baked wines.

© 2014 Elsevier Ltd. All rights reserved.

1. Introduction

Flavour is one of the most significant factors of wine quality, determining the consumer acceptance or rejection. Generally speaking, the aroma of wines is influenced by several differing compounds, originated from grapes or resulting from winemaking, ageing and storage. In fact, these compounds act as a fingerprint of each type of wine. In some cases, the occurrence of a particular compound is enough to give the characteristic aroma of a wine (Polaskova, Herszage, & Ebeler, 2008).

Madeira wine is a well-known fortified wine distinguished for its superior quality and it is characterised by marked and intense flavours (V. Pereira, 2011). Its winemaking can include a peculiar maturation process, a heating step known as *estufagem*. This step consists of heating the fortified wine to about 45 °C, for at least 3 months. Then, the oxidative ageing goes further, since wine is placed in wooden casks (regularly used casks), for at least 3 years. A premature ageing takes place along with heating, being acquired some of the aroma characteristics considered typical of the finest Madeiras (older wines only matured in oak casks for several years).

* Corresponding author at: Centre of Exact Sciences and Engineering, University of Madeira, Campus da Penteada, 9000-390 Funchal, Portugal. Tel.: +351 291705122.

E-mail address: vpereira@uma.pt (V. Pereira).

It is well known that heating promotes important changes in the aroma of foods. Up to date, there are some studies dealing with the accelerated oxidative ageing, promoted by thermal processing, on the volatile profile of wines. Deibner and Bernard, in 1956, as referenced by Cutzach, Chatonnet, and Dubourdieu (1999), studied the effect of heat treatment on wine, pointing out the important role of Maillard reactions in the formation of the aroma of thermally processed sweet fortified wines, but at that time, they were unable to identify the respective compounds. Cutzach et al. (1999) investigated the ageing of red and white sweet fortified wines (*Vins doux Naturels*) following an experimental laboratory study, in which, wines were forced-aged through heating at 37 °C for 12 months. Among the developed molecules during the accelerated ageing, the same authors found that sotolon (3-hydroxy-4,5-dimethyl-2(5H)-furanone), 5-ethoxymethylfurfural, 5-hydroxymethylfurfural (HMF), furfural acetylformoin and hydroxymaltol were involved in the aroma of sweet fortified wines. Sotolon has also been considered as a key odorant of the typical aroma of oxidative aged Port wine (Silva Ferreira, Barbe, & Bertrand, 2003). Escudero, Cacho, and Ferreira (2000) also performed studies dealing with wine oxidative ageing, through laboratory-oxidised samples at 20 °C for several weeks. According to olfactometric studies (GC–O analysis), they found that the impact odorants of oxidised white wines were essentially 2,4,5-trimethyldioxolane, methional, sotolon and eugenol. Changes in

the volatile content of Fino Sherry wines, exposed to heating (45 °C) and UV-Vis radiation, have also been reported (Benítez, Castro, Natera, & Barroso, 2006). These experiments revealed the decrease of most esters, acids and alcohols, and the increase of furfural and benzaldehyde. López de Lerma, Peinado, Moreno, and Peinado (2010) thermally processed sweet Pedro Ximénez wines at 65 °C for up to 30 days and reported the increase of volatile Maillard products, specifically of HMF, 5-ethoxymethylfurfural, dihydromaltol, 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one (DDMP), 2-methyltetrahydrofuran-3-one, furaneol, dihydro-2-methyl-3(2H)-furanone and cyclotene. Loscos, Hernández-Orte, Cacho, and Ferreira (2010) reported that accelerated ageing, at 50 °C for 9 weeks, of wines supplemented with grape flavour precursors also introduced important changes in the volatile composition. The same study showed that the main differences were observed in the first week of accelerated ageing. Most compounds first showed a significant increase and later a steady reduction, including Riesling acetal, 1,1,6-trimethyl-1,2-dihydronaphthalene (TDN), and (*E*)-1-(2,3,6-trimethylphenyl)buta-1,3-diene (TPB). However, vanillin derivatives, furan linalool oxides, 3-oxo- β -ionone, actinidols, 4-ethylphenol, and guaiacol showed a continuous increase during the ageing process.

Fedrizzi et al. (2011) studied the effect of accelerated ageing (30 °C for 60 days) on the fermentative, varietal and sulfur compounds of a dry red Botrytis Amaron wine and found out that benzaldehyde, phenylacetaldehyde, syringaldehyde and vanillin showed a perceptible increase, while several sulfur compounds disappeared after just 15 days, under oxidative conditions. They also tentatively identified methionol-S-oxide for the first time in this kind of wine. More recently, Cejudo-Bastante, Hermosín-Gutiérrez, and Pérez-Coello (2013) also compared accelerated ageing (50 °C during 7 days) with conventional storage (18 °C) analysing the volatile composition of Chardonnay white wines. They concluded that dioxanes, dioxolanes, and TDN were developed while some alcohols, terpenes, and furanic compounds disappear with accelerated ageing. Moreover, their results demonstrated that β -damascenone and TDN levels were quite higher in the accelerated-aged wine.

In the case of Madeira wines, which are traditionally forced-aged, few studies were done regarding the effect of the baking step on their volatile profile. The first study, carried out by Oliveira e Silva et al. (2008) was based on GC-O analysis and highlighted the occurrence of particular volatiles that imparted notes considered typical of finest Madeiras bouquet. Those volatiles were identified as sotolon, furfural, 5-methylfurfural, 5-ethoxymethylfurfural, methional, and phenylacetaldehyde.

The aim of the current work is to study the impact of *estufagem* on the global GC-MS volatile profile of Madeira wines, focusing to a large extent on individual volatile compounds, namely some that are barely reported in Madeira wines, in order to highlight the global ageing process. For this purpose, three young Madeira wines: dry Tinta Negra (TN, red variety), sweet TN and sweet Malvasia (white variety) were heated at 45 °C for 3 months. Additionally, overheating conditions, 70 °C for 1 month, were also studied to force the development of heating-specific volatiles.

2. Experimental

2.1. Wine samples

Three Madeira wines from two *Vitis vinifera* L. grape varieties of 2007 harvest were studied: dry TN, sweet TN and sweet Malvasia. The wines were produced using the winemaking practices of a Madeira wine producer. The elaboration of these wines was conducted in separated stainless steel tanks. The alcoholic fermenta-

tion was conducted under controlled temperature and without adding any commercial yeast. The fermentation for TN sweet wine was stopped adding alcohol (95% v/v) when the must specific gravity attained 1.025 g/mL, which corresponded to 115 g/L of reducing sugars in the wine, while for TN dry it was allowed to reach a specific gravity of 0.986 g/mL before fortification, allowing a low level of residual sugars in the wine (about 4 g/L). Malvasia sweet wine must was fermented up to 1.019 g/mL, keeping the reducing sugars at 96 g/L. After post-fermentation treatments, each wine was heated at 45 °C for 3 months in a special pilot scale system, equipped with 200-L stainless steel tanks, fitted with heat coils that allow hot water to circulate inside the container. To force the development of volatiles specific from overheating, each wine was also submitted to 70 °C for 1 month (overheating conditions). All samples were kept at -20 °C before being analysed.

2.2. Chemicals

All reagents were of analytical grade. Dichloromethane HPLC-grade was from Fisher Scientific (Loughborough, UK) while absolute ethanol was supplied by Sigma-Aldrich (St. Louis, MO). Ultra-pure water was obtained from a Milli-Q system (Millipore, Milford, MA). Solid anhydrous sodium sulfate was from JMGS (Lisbon, Portugal) while the 3-octanol standard was from Sigma-Aldrich. LiChrolut EN resin was supplied by Merck Co. (Darmstadt, Germany), while the 6-mL polypropylene cartridges and respective frits were obtained from Supelco (Bellefonte, PA). Solid-phase extraction was carried out in a 12-port Visiprep™ SPE vacuum manifold from Supelco.

2.3. Sample extraction

The screening of volatiles was accomplished based on the solid-phase extraction (SPE) method proposed by López, Aznar, Cacho, and Ferreira (2002). Briefly, 120 mg of LiChrolut EN resin were packed in a 6-mL cartridge. After conditioning the resin, in the SPE station, with 4 mL of dichloromethane, 4 mL of methanol and 4 mL of ethanolic solution (18%, v/v), 50 mL of wine spiked with 25 μ L of 3-octanol (491 mg/L) were passed through the resin at about 2 mL/min. Then, the sorbent was dried by passing a small flow of air through it for 15 min. Finally, wine volatiles were eluted with 1.3 mL of dichloromethane and the extract dried with sodium sulfate, being kept at -20 °C until analysis. All samples were extracted in duplicate.

2.4. GC-MS analysis

The extracts were analysed using a TRACE GC Ultra gas chromatograph equipped with the ISQ single quadrupole (electron impact mode) and the TriPlus autosampler (liquid mode) from Thermo Scientific (Hudson, NH). One microlitre of extract was vaporised in the injector port at 230 °C in splitless mode (1 min). All extracts were injected twice. The column was a DB-WAXetr 30 m \times 0.32 mm with 0.5 μ m film thickness from Agilent J&W (Folsom, CA) and the carrier gas was He at 1 mL/min. The ionisation voltage was 70 eV with transfer line and ion source temperatures kept at 230 and 240 °C, respectively. The oven temperature program started at 40 °C for 5 min then increased up to 230 °C at 3 °C/min and finally was kept at 230 °C for 15 min. The *m/z* 30–400 mass range was recorded.

The identification of compounds was made by comparison of the mass spectra obtained with those present in NIST08 and Wiley 6.0 MS library databases, and comparing the obtained Kovats indexes with those stated on NIST Chemistry WebBook (Stein, 2008). The compounds, namely those that were only identified comparing the obtained mass spectra with those present in the

Download English Version:

<https://daneshyari.com/en/article/1183861>

Download Persian Version:

<https://daneshyari.com/article/1183861>

[Daneshyari.com](https://daneshyari.com)