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Impact of storage time and temperature on furanic derivatives formation in wines using microextraction by packed sorbent tandem with ultrahigh pressure liquid chromatography

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ABSTRACT

This paper reports the effect of storage temperature, storage time and glucose content on furanic derivatives (FDs) formation in fortified Madeira wines using headspace solid phase microextraction (HS–SPME) combined with gas chromatography-quadrupole mass spectrometry (GC–qMS), and microextraction by packed sorbent (MEPS) tandem with ultrahigh pressure liquid chromatography coupled to photodiode array detection (UPLC–PDA). Based on the results obtained, MEPS/UPLC–PDA showed better FDs extraction compared to HS–SPME/GC–qMS, in terms of analysis reduction, sensitivity, reproducibility and allow detect all target furanic derivatives considered in this study. 5-Hydroxymethyl-2-furfural, one of the most abundant FDs in wines, is not detected by HS–SPME/GC–qMS, which suggest that this methodology is not the most suitable to achieve the purpose of this study.

Regarding the impact of storage time and temperature on FDs formation using MEPS/UPLC–PDA, it was verified an increase on FDs concentration during storage time independently of storage temperature as well as glucose content, and this increase is also favored by temperature increases, being more significant at 55 °C. The results obtained in this study could be used as a useful tool in winemaking field in order to introduce changes in fermentation as well as baking (*estufagem*) process, and/or predict the effects of storage time by application of high temperatures.

1. Introduction

Wine, a widely consumed beverage throughout the world, is a very complex matrix composed mainly by water, alcohol and sugars; additionally, it contains a wide range of minor organic and inorganic constituents. The concentration levels of these compounds are significantly influenced by many factors including grape variety, climate, wine-growing area and the winemaking process.

Madeira wine is a fortified wine produced in the Madeira Island, Portugal, characterized by supplementation with distilled spirit of grape origin at different fermentation stages to obtain an alcoholic level between 18 and 21% (v/v) and different glucose levels ranging from dry (till 25 g/L, fermented to low sugar levels) to sweet (130 g/L, partial fermentation) wines. Then, the wine is submitted to a baking process called as "*estufagem*", i.e., the wine is placed in large coated vats and the temperature is slowly increased at about 5 °C per day and maintained at 45–50 °C for 3 months. After this treatment, the wine is allowed to undergo a maturation process in oak casks for a minimum of 3 years. Finally, some Madeira wines suffer an ageing process, from minimum of 3–20 years or longer, in cellars at 30–35 °C by sun influence, and a humidity degree higher than 70% (Pereira, Reis, Saraiva, & Marques, 2010a; Perestrelo, Petronilho, Câmara, & Rocha, 2010; Perestrelo, Silva, & Câmara, 2015). During "*estufagem*" complex reactions occur as the result of several underlying physical, chemical and biochemical mechanisms promoted dough heating, which are essential for the development of the typical aroma, taste and color of Madeira wines (Pereira, Albuquerque, Ferreira, Cacho, & Marques, 2011; Perestrelo, Albuquerque, Rocha, & Câmara, 2011, chap. 7; Perestrelo et al., 2015). Among the formed compounds, furanic derivatives (FDs) are a class of heterocyclic compounds that result during nonenzymatic browning reactions, which are responsible for food organoleptic properties (Pereira et al., 2010a, Pereira, Reis, Saraiva, & Marques, 2010b; Pereira et al., 2011; Perestrelo et al., 2015) (see Fig. 1). Literature data showed there are several sources and mechanisms of FDs formation during thermal processing such as

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thermal degradation of carbohydrates or Maillard reactions of reducing sugars with amino acids, thermal oxidation of polyunsaturated fatty acids and ascorbic acid decomposition (Alañón, Rubio, Díaz-Maroto, & Pérez-Coello, 2010; Nie, Huang, Hu, Zhang, Wang, Li, et al., 2013; Petisca, Henriques, Pérez-Palacios, Pinho, & Ferreira, 2013). The presence of FDs in wine leads to changes in the flavor, color, aroma composition and organoleptic characteristics. Regarding to FDs toxicity, recent studies have shown that 5-hydroxymethyl-2-furfural (5HMF) derivatives, such as 5-chloromethyl- and 5-sulfoxymethylfurfural (SMF), have been associated with cytotoxic, genotoxic, and tumoral effects. Furthermore, 5-methyl-2-furfural (5 MF) and 2-furfural (F), have also been reported with genetic toxicity (mutagenicity) by reaction with DNA (Hu, Hernandez, Zhu, & Shao, 2013; Pereira et al., 2011; Zirbes et al., 2013). By the other hand, FDs also could be used as potential wine aging markers which is important to detect frauds and to ensure the authenticity of the wine (Perestrelo, Barros, Rocha, & Câmara, 2011). Moreover, the literature shows that the temperature reduction in the thermal treatment is the first option to minimize the FDs formation, and it can be achieved by heating at low temperatures and pressures for longer times (Aguilar, Garvin, Azuara, & Ibarz, 2015). Consequently, the determination of FDs in wine is very important for the control of those compounds.

Several methods have been reported for FDs determination in different matrices (e.g. wine, coffee, honey, bread, vinegars) using solid phase microextraction (SPME) combined with gas chromatography-mass spectrometry (GC-MS) (Bröhan, Huybrighs, Wouters, & Van der Bruggen, 2009; Perestrelo, Barros, Câmara, & Rocha, 2011; Petisca et al., 2013), as well as solid phase extraction (SPE) and microextraction by packed sorbent (MEPS) combined with high performance liquid chromatography (HPLC) or ultrahigh performance liquid chromatography (UPLC) (Hu et al., 2013; Perestrelo et al., 2015; Serra-Cayuela et al., 2013; Zhang, Wei, Liu, Lin, & Yuan, 2014). In addition, SPME and MEPS are considered an ideal sample preparation since they showed several features such as (i) minimal sample loss and a maximum recovery of the target analyte, (ii) elimination of coexisting components with high yield, (iii) simple, fast and inexpensive method, (iv) adapted with analytical instruments, and (v) in conformity with green chemistry (Pereira, Gonçalves, Alves, & Câmara, 2013; Pereira, Silva, Perestrelo, Gonçalves, Alves, & Câmara, 2014).

For this reason, the main goal of this study was to evaluate the impact of storage time (control (day 0) to 100 days) and temperature (45, 50 and 55 °C) in 5-hydroxymethyl-2-furfural (5HMF), 2-furfural (F), 2-furyl methyl ketone (FMK), 5-methyl-2-furfural (5 MF) formation in fortified wine with different glucose content, 135 (sweet) and 55 (dry) g/L. The FDs were extracted using HS-SPME and MEPS and analyzed by GC-qMS and UPLC-PDA, respectively.

2. Material and methods

2.1. Chemicals and materials

Furanic derivatives, such as 2-furaldehyde (F, 99%), 5-methyl-2-furaldehyde (5 MF, 99%), 2-furyl methyl ketone (FMK, 99%), 5-hydroxymethyl-2-furaldehyde (5HMF, 99%), formic acid (FA, > 98%), acetonitrile (ACN, 99.5%) and methanol (MeOH, 99.5%) were purchased from Sigma–Aldrich (St. Louis, MO, USA). All these chemicals were analytical gradient grade and used as received. Ultrapure water was purified with a Milli-Q ultra-pure water system from (Millipore, Milford, USA).

The MEPS gas-tight syringe (250 μL) and the BIN (Barrel Insert and Needle) containing the sorbent material were from SGE Analytical Science (Melbourne, VIC, Australia). The SPME holder for manual sampling and 75 μm carboxen-poly(dimethylsiloxane) fibre were purchased from Supelco (Aldrich, Bellefonte, PA, USA).

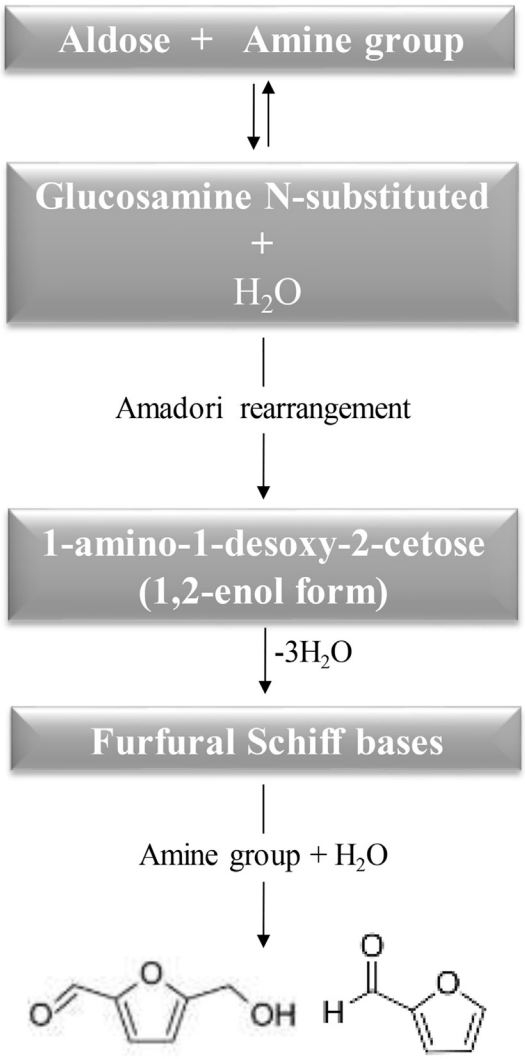

Fig. 1. Formation furanic derivatives pathways.

A HANNA instruments pH209 pH meter (Woonsocket, USA) was used to pH adjustments. The Acquity CORTECS UPLC® C18 analytical column (2.1 mm × 100 mm, 1.6 μm particle size) was purchased from Waters (Waters, Milford, MA, USA). Chromatographic mobile phases were prepared with ultra-pure water and LC-gradient grade methanol (MeOH). Before injection all eluents and extracts were filtered through 0.22 μm PTFE membrane filters from Pall Corporation (Ann Arbor, MI, USA) to remove any impurities.

2.2. Wine samples

Twenty monovarietal fortified Madeira wines obtained from Malvazia (sweet wine, n = 10) and Sercial (dry wine, n = 10) were considered in this study. The samples were kindly provided by Madeira Wine Company, the most representative producer of Madeira wine, transported to the laboratory in commercial 750 mL glass bottles and stored in the dark at 4 °C until analysis. The ethanol content of the Madeira wines under study ranged from 18 to 21% (v/v).

Afterwards, 50 mL of wine samples were put in 100 mL brown bottles, in a total of 22 bottles, and were tightly capped. They were