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Aroma release in the oral cavity after wine intake is influenced by wine matrix composition

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ABSTRACT

The aim of this study has been to investigate if wine matrix composition might influence the interaction between odorants and oral mucosa in the oral cavity during a "wine intake-like" situation. Aroma released after exposing the oral cavity of three individuals to different wines (n = 12) previously spiked with six target aromas was followed by an -in vivo intra-oral SPME approach. Results showed a significant effect of wine matrix composition on the intra-oral aroma release of certain odorants. Among the wine matrix parameters, phenolic compounds showed the largest impact. This effect was dependent on their chemical structure. Some phenolic acids (e.g. hippuric, caffeic) were associated to an increase in the intra-oral release of certain odorants (e.g. linalool, β-ionone), while flavonoids showed the opposite effect, decreasing the intra-oral release of aliphatic esters (ethyl hexanoate). This work shows for the first time, the impact of wine composition on oral-mucosa interactions under physiological conditions.

1. Introduction

Many works in the literature have been focused in the analytical determination of impact aroma compounds through olfactometric techniques (Munoz-Gonzalez, Rodriguez-Bencomo, Victoria Moreno-Arribas, & Angeles Pozo-Bayon, 2011; Robinson et al., 2014). Although odor impression is the first step on wine aroma perception (orthonasal perception), this process continues during the oral phase of consumption when the wine is ingested (retronasal perception). This second mechanism of aroma perception only occurs when the velum tongue border is open and the odorants are transported by the swallowing breath into the nasal cavity (Buettner, Beer, Hannig, & Settles, 2001). The retronasal impressions can be divided in the immediate aroma impression when the wine has been just swallowed, and the prolonged retronasal aroma perception after swallowing, called after smell or after-odor, which is responsible for the long lasting perception of aroma compounds (aroma persistence) (Buettner et al., 2001). For the aroma persistence takes place, aroma compounds have to be adsorbed to oral and pharyngeal mucosa, forming food aroma depots and/or food coatings (Buettner, 2004). The capacity of aroma molecules to interact with oral mucosa might then depend on the physicochemical characteristics of the aroma compounds, but also on the composition of the wine matrix itself and on the human physiology (e.g. saliva composition) (Pozo-Bayón, Muñoz-González, & Esteban-Fernández, 2016; Ployon, Morzel, & Canon, 2017). Therefore, aroma persistence might be impacted by both, the adsorptive capacity of odorants and matrix components to interact with oral mucosa and the degree of release of the aroma compounds from these depots.

The impact of the aroma compound type (structure and physicochemical characteristics) on its adsorptive capacity to bind into the oral mucosa during a simulated wine consumption situation has been recently investigated. In this work, Esteban-Fernandez an collaborators (Esteban-Fernández, Rocha-Alcubilla, Muñoz-González, Moreno-Arribas, & Pozo-Bayón, 2016) developed an *-in vivo* intraoral SPME methodology to monitor the aroma released from oral mucosa after the exposure of wine to the oral cavity. In these conditions, the human mouth resembled a close chamber (velum closed), in which the nasal and oral cavities are not communicated, allowing to have a good picture of the intra-oral aroma released from oral mucosa (Buettner et al., 2001). Using this approach, differences on aroma release kinetics among chemically different odorants over five minutes after spitting out the wine were observed. Some aroma compounds, such as esters, showed a very fast release in the first seconds after rinsing but also a rapid drop in their concentration, indicating a low persistence for these typical wine aroma compounds. Contrarily, other compounds, such as β-ionone or guaiacol showed a different release pattern, with a

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progressive and slow release from oral mucosa after wine rinsing, thus, indicating a high oral persistence.

Although this behavior seems to be tightly linked to physico-chemical aroma characteristics, non-volatile wine matrix components might also impact the aroma binding capacity of wine odorants to oral mucosa. Previously, Buettner (2004) found differences on intra-oral aroma release behavior after the consumption of two different types of Chardonnay wines, which were explained by differences in wine matrix composition. In this regard, some wine matrix components such as phenolic compounds can bind to oral surfaces like teeth enamel and oral soft tissues (checks) (Gibbins, Proctor, Yakubov, Wilson, & Carpenter, 2014; Ginsburg, Koren, Shalish, Kanner, & Kohen, 2012; Payne, Bowyer, Herderich, & Bastian, 2009), salivary proteins (PRPs) (Canon et al., 2013; McRae & Kennedy, 2011; Soares, Brandão, Mateus, & De Freitas, 2017), or oral epithelial cells (Payne et al., 2009). In this regard, these interactions between phenolic compounds and oral components could modify the rate of interactions between aroma compounds and oral mucosa (e.g. competence phenomenon). Besides of this, different works in the literature have described interactions among aroma molecules and wine matrix components using different *-in vitro* approaches (Dufour & Bayonove, 1999; Genovese, Piombino, Gambuti, & Moio, 2009; Jung & Ebeler, 2003; Mitropoulou, Hatzidimitriou, & Paraskevopoulou, 2011; Robinson, Ebeler, Heymann, & Trengove, 2009; Rodríguez-Bencomo et al., 2011; Villamor, Evans, Mattinson, & Ross, 2013). Therefore, it is likely that these interactions might also happen in oral conditions modifying the rate of retention/release of aroma compounds from the oral mucosa and ultimately wine aroma persistence.

Nonetheless, the role of specific wine matrix components on aroma-oral mucosa interactions and their impact on aroma release is scarcely known and it has been only investigated by sensory studies (Baker & Ross, 2014b; Goodstein, Bohlscheid, Evans, & Ross, 2014; Sáenz-Navajas et al., 2010). In these studies, well-designed experiments using different types of wines spiked with non-volatile components and some sensory approaches involving time-intensity assays, have allowed to conclude that differences in wine matrix composition might correlate with differences in aroma persistence.

Therefore, the aim of this work was to gain an insight into the chemical mechanisms determining interactions between aroma compounds and wine matrix components into the oral cavity during a "wine intake-like" situation. In this study, intraoral aroma release data (mean of 3 panelists) were obtained by an *-in vivo* SPME methodology using twelve different commercial wines produced under different wine-making technologies and therefore, with different matrix composition. All the wines were adjusted to the same ethanol content and aromatized with six target aroma compounds from different chemical classes. The chemical matrix composition was determined and intra-oral aroma release data were correlated to wine compositional parameters by using different chemometric tools.

2. Materials and methods

2.1. Wine samples

Twelve Spanish commercial wines (four whites, two rosés and six reds) representative of different winemaking technologies were selected for this study (Table 1). In order to minimize the effect of ethanol on the volatility of aroma compounds, all the wines were adjusted to the same ethanol concentration (14.5% v/v) using food grade ethanol (Panreac Química S.A. Barcelona, Spain). To reinforce the aroma profile and to facilitate oral aroma monitoring independently of their endogenous presence in the commercial wines, all the wines were spiked with six target food-grade aroma compound from Sigma-Aldrich (Steinheim, Germany) characterized by presenting different physicochemical properties (Table 2).

To do that, six independent aroma stock solutions were prepared in ethanol absolute. From here, each aroma compound was added to the wines to obtain a final concentration of 1 mg L^{-1}. Previous to the oral aroma monitoring, a static headspace SPME sampling procedure already described (Rodríguez-Bencomo et al., 2011), was used to determine the total aroma released in the wines with and without added aroma. This step allowed us to normalize the data and to calculate the percentage of aroma coming from the aromatization process in all the wines, by applying the equation below, where RPA corresponds to the relative peak area (peak area compound/peak area internal standard) using the static headspace SPME sampling procedure:

$$\%\ endogenous\ aroma = \frac{Total\ amount\ of\ aroma\ (RPA)\ in\ the\ original\ wine}{Total\ amount\ of\ aroma\ in\ the\ aromatised\ wine\ (RPA)} * 100$$

This value was used to correct for differences in the aromatized wines among the wines used in this study (Table S1). Although this experimental approach requires the correction of the data for endogenous aroma compounds, it avoided the manipulation of the original wine sample reducing the safety hazards associated to the use of organic solvents to remove endogenous wine aroma compounds.

2.2. Intra-oral SPME sampling of odorants

2.2.1. Volunteers

Three volunteers (females) between 24 and 41 years old previously trained in the intraoral SPME procedure participated in this study. They were instructed not to eat, drink or smoke 2 h before the experiments. They had no known illnesses and had self-reported normal olfactory and gustatory functions. They were informed of the nature of this study and gave their writing consent to participate.

2.2.2. Intra-oral SPME procedure

Fifteen minutes before each experiment, the volunteers had to clean their mouths and rinse them with a bicarbonate solution and water in order to have the most similar "oral status". The intra-oral SPME procedure previously described (Esteban-Fernández et al., 2016) was used to monitor the aroma released from oral mucosa. Briefly, fifteen ml of the aromatized wines were taken into the oral cavity, kept for 30 s in order to favor the equilibration of the aroma compounds within the oral cavity and spat-off. During rinsing, special care was taken to keep the lips closed, not to swallow and not to open the velum – tongue border prior to expectoration. At 30 s after expectoration, a DVB/CAR/PDMS (Divinylbenzene/Carboxen/Polydimethylsiloxane 50/30 µm thickness -2 cm length-) coated SPME fiber (Supelco, Bellefonte, PA) with a home-made adaptor consisting in a plastic changeable tube inside a septum in which the SPME fiber was placed through, was sited into the oral cavity of the panelist. After 2 min of intra-oral aroma extraction, the fiber was removed from the oral cavity, and immediately placed into the split/splitless injector of the Gas Chromatograph (GC) (Agilent 6890 N) coupled to a quadrupole Mass Detector (MS) Agilent 5973. Analyses were performed three times with each wine by the three volunteers (12 wines × 3 volunteers × 3 repetitions = 108 injections).

2.2.3. GC/MS analysis

Desorption of the oral aroma extracts adsorbed to the fiber was performed in the injector of the GC–MS system in splitless mode for 1.5 min at 270 °C. After each injection the fiber was cleaned for 10 min to avoid any memory effect. Volatile compounds were separated on a DB-Wax polar capillary column (60 m × 0.25 mm i.d. × 0.50 µm film thickness) from Agilent (J & W Scientific, Folsom, USA). Helium was the carrier gas at a flow rate of 1 mL min^{-1}. The oven temperature was initially held at 40 °C for 2 min, then increased at 8 °C min^{-1} to 240 °C and held for 15 min.

For the MS system (Agilent 5973 N), the temperature of the transfer line, quadrupole and ion source were 270, 150 and 230 °C respectively.