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Intra-oral adsorption and release of aroma compounds following in-mouth wine exposure

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ABSTRACT

Wine "after-odour" defined as the long lasting aroma perception that remains after wine swallowing is an outstanding characteristic in terms of wine quality but a relatively unstudied phenomenon. Among the different parameters that might affect wine after-odour, the adsorption of odorants by the oral mucosa could be important but has been little explored. In this work, the impact of the chemical characteristics of aroma compounds on intra-oral adsorption was assessed by an *in vivo* approach that determined the amounts of odorants remaining in expectorated wine samples. In addition, the subsequent aroma release after in-mouth wine exposure was studied by means of intra-oral SPME/GC–MS using three different panellists. Oral adsorption of the aroma compounds added to the wines ranged from 6% to 43%, depending on their physicochemical characteristics. A progressive intra-oral aroma decrease at different decay rates depending on compound type and panellist was also found. The strength of the aroma–oral mucosa interactions seems to explain these results more than the amount of compound adsorbed by the oral mucosa.

1. Introduction

In recent years, a lot of research has been addressed toward understanding retronasal aroma perception because it is a key modulator for food consumption and consumer preferences (Gierczynski, Guichard, & Laboure, 2011; Mishellany-Dutour et al., 2012). The perception of aroma during food consumption is a complex phenomenon involving the release of odorants within the oral cavity from the food material, their transport *via* the retronasal route to the nasal cavity, followed by their interaction with the respective receptors in the olfactory epithelium and the subsequent transduction of the sensory signals to the brain. During food consumption, two key modes of aroma release and perception have to be distinguished: the immediate aroma impression, produced when a liquid or solid food is just swallowed, and the prolonged retronasal aroma perception after swallowing, often called the "after-odour" or aroma persistence (Buettner, 2004).

The long lasting aroma perception of wine odorants after swallowing is a feature of special importance during wine tasting that serves to assess wine quality. The after-odour is also included in the wider term "wine finish" defined by Jackson (2002) as the lingering flavour, taste and mouthfeel that one observes after the swallowing or expectoration of wine (Baker & Ross, 2014). A recent work has however highlighted the idea that, in spite of its importance to assess wine quality, the term "wine finish" is a relatively unexplored aspect compared to other wine sensory attributes such as aroma and flavour (Baker & Ross, 2014). In fact, only recent publications have addressed this subject and they have been performed following sensory approaches through the use of well-designed time–intensity studies using white and red wine models supplemented with different matrix components and several types of odorant compounds (Baker & Ross, 2014; Goodstein, Bohlscheid, Evans, & Ross, 2014). In model white wines the length of "finish" of specific aromas is highly dependent on the type of odorant present in the system; for instance, some "fruity" notes finished earlier than "coconut", "floral" and "mushroom" notes. Apart from the type of odorant, other factors such as the coexistence of different odorants in the wine (Goodstein et al., 2014) or the presence of other matrix components, such as ethanol and tannins, can affect the intensity and length of the aroma (Baker & Ross, 2014).

To understand why these molecules present different rates of "finish", it is important to take into account their interaction with human physiology. In fact, the higher or lower adsorption capacity of odorant compounds by oral and pharyngeal mucosa has been described as an additional physiological mechanism to explain

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the after-odour impression following food swallowing (Buettner, Beer, Hannig, & Settles, 2001; Buettner & Mestres, 2005; Lasekan, Buettner, & Christlbauer, 2009). In some of these works, the oral adsorptive potency of some odorants contained in water solutions was proven by quantifying the remaining amounts of specific odorant compounds after sample expectoration (Buettner, 2002; Buettner et al., 2001). Nonetheless, there is no previous evidence on the adsorptive potency of odorants to the oral cavity following wine exposure. Only one previous study has shown a positive correlation between the amount of aroma released from the oral cavity after wine expectoration and the long lasting intensity of specific aroma nuances (Buettner, 2004). In this work, authors used an interesting analytical approach based on the application of stir-bar sorptive extraction (SBSE) (Baltussen, Sandra, David, & Cramers, 1999) to monitor intra-oral aroma release after the exposure of the oral cavity to two types of white wines using a PDMS-coated stir bar. Although very valuable, this study was performed in "real" wines, characterised by a complex volatile profile that could contain many hundreds of different types of aroma compounds at different concentrations (Robinson et al., 2009), which also might exhibit differences in matrix composition (polyphenols, proteins, etc.), and other oenological parameters (pH, alcohol content, etc.). All of these factors might affect the partition coefficients and mass transfer of aroma compounds between the liquid and gas phases and therefore modify intra-oral aroma release. Many works in the field using *in vitro* static or dynamic headspace conditions have already underlined the importance of aroma–wine matrix interactions to determine the extent of aroma release (Rodriguez-Bencomo et al., 2011; Jung and Ebeler,2003; Robinson et al., 2009, Villamor, Evans, Mattinsonc, & Ross, 2013; Dufour & Bayonove, 1999). More recently, Muñoz-González, Martín-Álvarez, Moreno-Arribas, and Pozo-Bayón (2014) using an *in vivo* approach have shown the impact of wine matrix composition on aroma release using aromatised wines.

These observations highlight the necessity of complementary scientific approaches taking into account more controlled wine systems and human physiology. This should allow more straightforward conclusions on the relationship between odorant structure and oral adsorptive and release capacity, to gain insight into the molecular mechanisms behind wine after-odour. Therefore, the objective of this work was to determine the impact of the type of aroma compound (physicochemical properties) on the oral adsorption and further release behaviour following in mouth wine exposure. To achieve this objective, a white wine was aromatised at the same concentration with a mixture of typical wine aroma compounds selected on the basis of their different physicochemical properties. Aroma adsorption into the oral cavity was determined by difference between the aroma added to the wine and that recovered in the expectorated sample after solvent extraction and subsequent GC–MS analysis. To monitor intra-oral aroma release, a modified HS-SPME technique (intra-oral SPME) was set up for this study and, once checked for its adequacy in terms of repeatability and response to increasing wine aroma concentration, it was used at different times after wine expectoration following an optimised consumption procedure. Finally, the aroma released from the oral cavity of three individuals after wine exposure was monitored at different times after wine expectoration (ranging from 30 to 300 s) to build intra-oral release profiles.

2. Material and methods

2.1. Wine samples

A low aromatic white wine from the Airen grape variety with pH 3.3, ethanol concentration 12% (*v*/*v*) and 278 mg gallic acid/L of total polyphenols (measured by the Folin–Ciocalteau assay) was selected for this study. Aromatisation was performed with a mixture of six food-grade aroma compounds (Sigma–Aldrich, Steinheim, Germany) representative of the wine volatile profile (ethyl hexanoate, β-ionone, linalool, guaiacol, β-phenylethanol and isoamyl acetate) and characterised for having a wide range of physicochemical properties (Table 1). For the aromatisation, six independent aroma stock solutions in food-grade ethanol (Panreac Química S.A., Barcelona, Spain) were prepared and from there, each aroma compound was added to the wines to obtain different concentrations depending on the experiment (0.5, 1, 1.5 or 2 mg/L).

2.2. Panellists

Three volunteers (females) between 24 and 40 years old previously trained in the intra-oral aroma trapping procedure participated in this study. Two of them also participated in the oral adsorption experiment. 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. All of them had brushed their teeth and fifteen minutes before each experiment, the panellists rinsed their mouths with water:bicarbonate solution first and then with tap water. The monitoring of the oral cavity of the panellists for the six compounds of interest was performed before each analysis. The sampling procedures were explained in detail to the subjects who provided written consent prior to participation.

2.3. Aroma adsorbed to oral mucosa

To determine the aroma adsorbed by the oral surface, the previously described Spit-Off Odorant Measurement procedure (SOOM) (Buettner & Schieberle, 2000) with some modifications was applied. Fifteen millilitres of the aromatised wine (1 mg/L of each aroma compound) were taken into the oral cavity, kept for

Table 1
Physicochemical properties of the aroma compounds employed in this study.

Compound	CAS number	MW[a] (g mol^{-1})	BP[b] (°C)	log *P*[c]	OT (μg/L)[d]	Descriptor[e]
Isoamyl acetate	123-92-2	130	134	2.26	30	Banana
Ethyl hexanoate	123-66-0	144	167	2.83	5–14	Apple peel, fruit
Linalool	78-70-6	152	204	3.38	2–25	Flower, lavender
Guaiacol	90-05-1	124	211	1.34	9.5–10	Spice, clove
β-Phenylethanol	60-12-8	122	224	1.57	14000–100000	Honey, spice, rose
β-Ionone	8013-90-9	192	262	4.42	0.09	Raspberry, violet, flower

[a] Molecular weight.
[b] Boiling point.
[c] log *P* = log of the water partition coefficient estimated from molecular modelling software EPI Suit (U.S EPA 2000–2007).
[d] Odor thresholds compiled in Francis and Newton (2008).
[e] From flavornet (http://www.flavornet.org; accessed October 2009) database, from NIST web chemistry book (2005) (http://www.webbook.nis.gov/chemistry).