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Influence of phenolic compounds on the sensorial perception and volatility of red wine esters in model solution: An insight at the molecular level

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ABSTRACT

Impact of (+)-catechin and gallic acid on sensory perception and volatility of isoamyl acetate, ethyl isobutyrate, ethyl butyrate and ethyl octanoate was investigated in model solutions, by means of triangle tests, detection threshold determination and HS-GC–MS analyses. Catechin significantly altered the sensory perception of most esters (ethyl isobutyrate, ethyl butyrate and ethyl octanoate) while gallic acid displayed no impact. Ethyl butyrate and ethyl octanoate odour thresholds doubled or tripled in the presence of catechin, underlining a retention impact of phenolic compounds in liquid matrix. The headspace analyses displayed a decrease only in ethyl octanoate volatility in presence of catechin, whereas no significant difference in other esters concentrations was observed. This study indicated that phenolic compounds have a variable impact on aroma compounds' volatility and their sensory perception. The polarity of phenolic and volatile compounds as well as their spatial conformation also appeared to influence the interaction strength.

1. Introduction

The sense of smell and detection of the aromas in wine is the primary means through which wine is tasted and evaluated. The proportion of aroma compounds smelled by tasters is mainly governed by their volatility and solubility, which means their partitioning between liquid and gas phases. However, variations in the effective concentration in the headspace above the wine are possible, influenced by other wine constituents present in the liquid medium. Indeed, recent works underlined the complexity of wine sensory perception through the powerful influence of the wine non-volatile matrix on odorants release (Robinson et al., 2011; Saenz-Navajas et al., 2010). Through an original methodology based on reconstituted wine samples, Saenz-Navajas and coworkers showed the existence and effects of physicochemical interactions strong enough to make a white wine aroma smell like a red wine and vice-versa. In particular, it was demonstrated that ethanol, glucose, polysaccharides (arabinogalactan, pectin), proteins and phenolic compounds influenced the partitioning of volatiles (Dufour & Bayonove, 1999a; Ebeler & Thorngate, 2009; Mitropoulou, Hatzidimitriou, & Paraskevopoulou, 2011; Robinson et al., 2009). Regarding the sensory consequences of interactions between volatile and wine matrix constituents, Jones and co-workers showed that several aroma attributes were significantly affected by proteins, alcohol and glycerol concentration (Jones, Gawel, Francis, & Waters, 2008). They underlined that most of the interactions affecting perceived aroma were strongest when volatile concentration was low and also observed that polysaccharides slightly suppressed the intensity of overall aroma while overall flavour intensity was positively influenced by glycerol.

Polyphenols, especially anthocyanins and tannins comprise a significant portion of the non-volatile matrix components of wines. Anthocyanins are the pigmented compounds responsible for red wine colour and are essentially located in grape skins. Proanthocyanidins include a large range of phenolic compounds constituted of flavan-3-ol monomer subunits. Their structures vary according to the nature of their constitutive subunits, the mean degree of polymerisation (mDP) and linkage position (Cheynier et al., 2006; Prieur, Rigaud, Cheynier, & Moutounet, 1994). They are extracted from seeds and skins during the wine-making process. Proanthocyanidins are of great importance to sensory red wine quality due to their astringent and bitter properties (Gawel, 1998; Peleg, Gacon, Schlich, & Noble, 1999). Their concentrations in red wine vary from 1 to 4 g/L according to grape variety and to an even greater extent, winemaking methods (Ribéreau-Gayon, Glories, Maujean, & Dubourdieu, 2006).

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Several studies have focused on interactions between aroma molecules and polyphenols and further consequences on the volatility and release of odorants in model solution. Using exponential dilution analysis and NMR spectroscopy, Dufour and Bayonove (1999b) underlined the existence of weak interactions between catechin and aroma compounds in model solution. They suggested that hydrophobicity acts as driving force for bimolecular aroma–phenolic interactions. The impact of aroma compound nature (esters, benzaldehyde, limonene) and polyphenol (catechin, tannin) was also highlighted in this work (Dufour & Bayonove, 1999b). Using headspace solid-phase microextraction combined with GC–MS quantification, Jung and Ebeler (2003) confirmed the catechin impact on volatility of some aroma compounds. For the assessed esters and aldehydes, interactions induced a decrease in volatility. On the other hand, an increase in volatility or "salting-out" phenomenon was recorded in the presence of a ketone (2-heptanone). This study proved that headspace solid-phase micro extraction could be another useful tool for the study of these interactions (Jung & Ebeler, 2003).

Regarding anthocyanins, phenol-based flavour compounds (vanillin, syringaldehyde) led to interactions through copigmentation. The main driving force was still ascribed to hydrophobicity (Dufour & Sauvaitre, 2000). Other studies carried out on gallic acid have evidenced that interactions were principally π–π stacking between the galloyl ring and the aromatic ring of an aroma compound but secondary hydrogen-bonding effects help to stabilise the complex and enhance the specificity (Aronson & Ebeler, 2004; Jung, de Ropp, & Ebeler, 2000).

Most of these studies have been based on analytical determination, but some have documented the sensory impact of these interactions. Aronson and Ebeler (2004) showed that gallic acid and naringin decreased the perceived intensity of 2-methylpyrazine and ethyl benzoate in aqueous solution. In the same study, when tannins were added to wines, a significant effect on flavour volatility was observed by GC analysis, but effects were less apparent by sensory evaluation. In a non-Sauvignon Blanc wine diluted by half, Lund and co-workers highlighted that catechin, caffeic acid and quercetin addition showed various effects on the key aroma compounds, such as isobutyl methoxypyrazine, 3-mercaptohexanol and ethyl decanoate (Lund, Nicolau, Gardner, & Kilmartin, 2009). Catechin and quercetin could largely suppress the perception of 3-mercaptohexanol while gallic acid showed the opposite effect. For methoxypyrazine and ethyl decanoate, the added phenols induced a decrease of their relative perception.

Regarding wine aroma, over 1000 volatiles, deriving from grapes, fermentation or ageing processes have been identified. Among them, esters quantitatively constitute the majority of the volatile component in red wines (typically present in approximately mg/L concentrations in wines) (Ebeler & Thorngate, 2009). They are formed during fermentation, including fatty acid ethyl esters and acetate esters, both of which contribute important fruity notes such as "red" and "blackberry aromas" to red wines (Escudero, Campo, Fariña, Cacho, & Ferreira, 2007; Pineau, Barbe, Van Leeuwen, & Dubourdieu, 2009).

Interactions between volatiles and phenolic compounds have been clearly identified from a chemical point of view, while their sensory impact remains confusing. Moreover, these interactions appear to be complex, impacted by the phenolic and aroma structures as well as the liquid matrix (water, model solution, white or red wine). In most of the previous data, assessed concentrations of aroma and polyphenols were really high, often far from those found in wines. Insight into molecular level in model solution is necessary. This work intends to assess the impact of individual phenolic compounds, such as (+)-catechin and gallic acid on the sensory perception and volatility of different fruity esters in solution, modeling red wine. A particular focus was to work at "real" concentrations, close to potential red wine concentrations.

2. Materials and methods

2.1. Chemicals

Deionised water was purified with a Milli-Q water system (Millipore, Bedford, MA). Tartaric acid, ethyl butyrate, isoamyl acetate, ethyl isobutyrate, ethyl octanoate, gallic acid, (+)-catechin were purchased from Sigma Aldrich (Saint Louis, MO). Ethanol from Scharlau (Sentmenat, Barcelona, Spain) was HPLC grade and twice-distilled in order to remove any odorant contamination.

2.2. Sensory analyses

All analyses were performed in model solution constituted of twice-distilled ethanol (12%, v/v), tartaric acid (5 g/L) at pH 3.5 (adjusted with NaOH). Twenty judges were selected on the basis of availability and interest. In order to guarantee the stability of volatile samples through the sensory analysis duration, 4-mL samples were presented in 30-mL bottles, according to Tempere et al. (2011). For all sensory analyses, the bottles were labelled with three-digit random codes and were presented after equilibration for at least 12 h at 20 °C, with a randomised arrangement across panellists. All experiments were performed in duplicate.

Triangle tests (ISO-4120, 2004) were first set up to investigate the perceived orthonasal differences between aroma compounds (ethyl butyrate, ethyl octanoate, isoamyl acetate, ethyl isobutyrate) alone in model solution and in model solution containing gallic acid or catechin. Different combinations were assessed with varying concentrations of aroma compounds and polyphenols. Impact of gallic acid and catechin was assessed at 250, 500 and 2000 mg/L. Ethyl butyrate, isoamyl acetate and ethyl isobutyrate were assessed at 100 and 200 μg/L while ethyl octanoate was used at 300 and 600 μg/L.

Then, the detection thresholds of the aroma compounds were determined in model solution with or without phenolic compounds at 2 g/L, with an ascending procedure (six concentrations) and the three-alternative forced choice presentation method (AFC) (ISO-13301, 2002). To compare data obtained under the same conditions, thresholds of aroma alone and aroma added with phenol were determined in the same session. For each concentration, subjects received a set of three bottles; two of them were blank samples (model solution or model solution added with phenolic compounds) and one contained the odorant dilution (positive sample). Sensorial analyses tested the whole series of dilution sets, by asking each assessor to first sniff each bottle in the prescribed order and then choose the spiked sample in each set of three bottles.

2.3. Data analysis

For triangle tests, the number of correct answers were summed and compared to the data of the binomial table (minimum number of correct responses needed to conclude that a perceptible difference exists based on a triangle test (ISO-4120, 2004)). If the number of correct responses was greater than or equal to the number given in the table (corresponding to the number of assessors and the α-risk level chosen for the test), it was concluded that a perceptible difference existed between the samples.

The detection threshold was defined as the concentration at which the probability of detection was 50%. This statistical value was determined according to the adaptation of the ASTM-E1432 method employed by Tempere et al. (Cometto-Muñiz & Abraham, 2008; Tempere et al., 2011). The concentration/response function