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The dynamics of aroma compound transfer properties in cheeses during simulated eating conditions

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ABSTRACT

In vivo aroma release during solid food consumption is a complex phenomenon that depends on food structure and composition, as well as on oral processing (combination of mastication and incorporation of saliva into the food product). The objective of this study was to understand and to predict the physico-chemical properties of aroma compounds through the dynamics of flavor release during in-mouth oral processing of food before bolus swallowing. Within this context, the evolution of two aroma compounds during bolus formation was explored by studying the two main properties that account for mass transfer: air/bolus partition and mass transfer coefficients. Four types of industrial cheese products (varying in fat and firmness) flavored with ethyl propanoate and 2-nonanone were chosen. Each matrix was mixed with various amounts of artificial saliva to mimic boluses at different stages of mastication. The air/bolus partition coefficient was determined by the static phase ratio variation method (PRV), while the mass transfer coefficient was obtained by non-linear regression from dynamic headspace experiments. Results showed that there is a dilution effect on the air/bolus partition coefficient and both a dilution and a product effect (firmness) on the mass transfer coefficient of ethyl propanoate in the bolus. These results were also validated with 2-nonanone for the low-fat cheeses.

1. Introduction

When a solid food product is eaten, oral breakdown processing is required for the formation of a bolus ready to be swallowed. For brittle solids, mastication leads to the fragmentation of food into small individual particles. For cohesive foods such as cheeses, the chewing action and the mixing with saliva soften the material and change its rheological properties (Salles et al., 2011). Recent studies (Prinz & Lucas, 1997; Tarrega, Yven, Sémon, & Salles, 2011; van Der Bilt, Engelen, Abbink, & Pereira, 2007; Yven, Culioli, & Mioche, 2005) have shown that saliva incorporation into the food bolus was an important factor in determining bolus texture and cohesion. More specifically, saliva plays a key role in parameters such as the plasticity, the good lubrication and the cohesiveness of the bolus, properties which trigger the reflex swallowing event (Woda, Mishellany, & Peyron, 2006).

Saliva also plays a key role in flavor release during food consumption mainly by means of dilution or interaction effects with food ingredients (Buettner, 2002a, 2002b; Poinot, Arvisenet, Grua-Priol, Fillonneau, & Prost, 2009). The influence of saliva on aroma release has been widely studied, both from *in vivo* and *in vitro* systems, with either real (Buettner, 2002a, 2002b; Genovese, Piombino, Gambuti, & Moio, 2009; van Ruth & Roozen, 2000) or artificial saliva (Friel & Taylor, 2001; Genovese et al., 2009), and on various foods: water model solutions (Buettner, 2002a; Friel & Taylor, 2001), oil model solutions (van Ruth, Grossmann, Geary, & Delahunty, 2001), vegetables such as pepper and beans (van Ruth, Roozen, & Cozijnsen, 1995; van Ruth, Roozen, Nahon, Cozijnsen, & Posthumus, 1996; van Ruth & Roozen, 2000), complex foods (Odake, Roozen, & Burger, 1998) and wine (Genovese et al., 2009). However, no general observation can be established because the effect of saliva may vary depending on the experimental conditions. It mainly depends on the aroma compound (Buettner, 2002a, 2002b), the artificial saliva composition (Friel & Taylor, 2001; Genovese et al., 2009; Odake et al., 1998; van Ruth et al., 1996; van Ruth et al., 2001) and the dilution ratio (van Ruth et al., 2001). It also depends on the matrix studied (van Ruth & Roozen, 2000) and the incubation time with the flavored food sample (from 1 min to 3 h, according to the literature).

In spite of variations during eating, saliva is mainly composed of water (99.5%), proteins (0.3%) including mucins and enzymes, and inorganic substances (0.2%) (van Aken, Vingerhoeds, & de Hoog, 2007). Different types of artificial saliva composition have been studied in the literature with *in vitro* experiments, and many publications (Genovese et al., 2009; Odake et al., 1998; van Ruth et al., 1996; van Ruth et al., 2001) refer to the composition proposed by van Ruth et al. (1995). The effect of the type of mucin, and of the presence of salt on flavor release from sucrose solutions have been investigated (Friel & Taylor, 2001). The authors found that the

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cheapest and most commonly used mucin (from pig stomach) was a suitable substitute for *in vitro* studies in terms of the effect on volatile release. Moreover, salivary salts seemed to cause a modification of the interactions between the aroma compound and the mucin by changing its conformational state. The importance of adding salts to artificial saliva was also highlighted by van Ruth et al. (2001), who found that saliva cannot be replaced by water without altering phase equilibriums (for water and oil model systems).

Concerning the impact of saliva/product ratio on flavor release, results found in the literature are dependent on the type of food studied. For very low-fat foods such as vegetables, saliva acted like water and very weakly affected the air/product partition coefficients (van Ruth et al., 1996). For emulsion systems, on the contrary, an effect of saliva dilution was found as a result of the different solubilities and affinities of the compounds for the oil and the saliva phases (van Ruth et al., 2001). The authors also concurred on the greater importance of the saliva/product ratio with regard to saliva composition.

Only a few studies have investigated the combined effect of mastication and of saliva on the release of aroma compounds from semi-solid or solid foods (Odake et al., 1998; Poinot et al., 2009; van Ruth et al., 1995; van Ruth & Roozen, 2000). Model mouth systems that mimic mastication were created to study flavor release from red bell peppers (van Ruth et al., 1995; van Ruth & Roozen, 2000). Results from mentioned studies show that during intra-oral processing, mastication and salivary volume increase affect aroma release in opposite ways, making it difficult to predict *in vivo* aroma release for solid foods.

So far, the development of artificial mouths and chewing machines, as well as the knowledge of the fundamental physics of mass transfer have provided useful data to help in the understanding of the interactions between flavor compounds and other components of the food. However, the prediction of the behavior of flavor compounds in food systems remains limited (Piggott, 2000).

Many mathematical models have been developed to help us increase our understanding of flavor release during food consumption. When considering a liquid or semi-liquid food, a change in composition of the bolus and a decrease in viscosity have to be considered due to the dilution effect. Recent models have succeeded in modeling flavor release from these liquid boluses, including physiological parameters such as breathing, swallowing and salivary flow rates (Doyennette, de Loubens, Déléris, Souchon, & Trelea, 2011; Normand, Avison, & Parker, 2004; Trelea et al., 2008). However, when considering a solid matrix, modeling requires detailed knowledge of the mastication process and product fragmentation, which is difficult to obtain experimentally. Existing mechanistic models for solid foods have focused on chewing gums or candies. de Roos and Wolswinkel (1994) developed a model to study the release of aroma compound from a chewing gum. The mastication process was considered as a series of successive extractions of the gum phase and computed to optimize the chewing gum flavoring. However, this model was limited by the fact that the mastication process for a chewing gum is not representative of the one applied for other solid foods and by the fact that the model does not take individual variations such as salivary flow rates and respiratory frequencies into account. Other authors (Hills & Harrison, 1995; Wright & Hills, 2003) attempted to build more complex models with candies. To mimic brittle food consumption, they included mastication patterns based on power law or on probabilistic model, respectively, but their approaches are not comparable to real *in vivo* mastication data. Moreover, their models did not take simultaneous fragmentation and dissolution of the product into saliva into account. The changing composition of the bolus over time is a critical phenomenon that leads to changes in physico-chemical properties of aroma compounds and therefore affects their release in the gaseous phase. Understanding the evolution dynamics of those physico-chemical properties during the mixing of the product with saliva is thus of great importance for building more robust models to predict flavor release.

Within this context, the present study aims at exploring the evolution of the air/bolus partition coefficient and of the mass transfer coefficient for two aroma compounds during bolus formation to better understand phenomena involved in flavor release during food consumption. A combined experimental and modeling approach was applied in this work.

2. Materials and methods

2.1. Cheeses

Four industrial cheese products (melt-cheese technology) with different compositions and textures (two fat levels and two firmness levels) were designed for this study. Textural properties were characterized by the critical strain and the stress at breakdown as described by C. Yven et al. (2010). Those rheological parameters were extracted from trials performed at 20 °C with a rotational viscosimeter Thermofisher — VT550 equipped with a vane geometry FL1000. Data related to cheese characteristics are summarized in Table 1. High-fat cheeses presented lower critical strain at breakdown than low-fat cheeses; and firm cheeses presented a high breakdown stress.

2.2. Aroma compounds

Ethyl propanoate and 2-nonanone (Aldrich, Germany) were used and chosen for their difference in physico-chemical properties, particularly in terms of volatility (air/water partition coefficient) and hydrophobicity (log P) (see Table 2). They are also commonly used in food flavoring, or naturally present, especially in cheeses (Wolf, Perotti, Bernal, & Zalazar, 2010). Cheeses were flavored with 25.0 ± 5.0 ppm (w/w) of ethyl propanoate and 6.3 ± 1.5 ppm (w/w) of 2-nonanone. The final concentration of aroma compounds within products was controlled during and after product manufacturing by gas chromatography after an extraction step (Likens Nickerson method).

2.3. Artificial saliva composition

Our experimental design did not allow us to use real saliva for *in vitro* measurements due to the difficulty to collect and manipulate real saliva without altering its composition or its properties (necessity to interrupt enzymatic reactions after specific times, use of chemical/thermal treatment to limit microbiological developments, etc.). Artificial saliva was therefore used for this study. Our principal concern was to select the main ingredients present in real saliva that could have an impact on flavor release and to maintain proportions between constituents that were close to those of real saliva.

The artificial saliva used consisted of three components: water, mucin (for its effect on saliva viscosity and its possible interaction with aroma compounds (van Ruth et al., 2001)) and salt (for its effect on the conformational state of mucin (Friel & Taylor, 2001)). Only NaCl was selected for the sake of simplicity, but the global conductivity of our artificial saliva was corrected to be equal to that of real saliva (Drago et al., 2011). The composition of the artificial saliva was: 0.185 g of NaCl (GRP RECTAPUR, VWR INTERNATIONAL), 0.216 g of mucin (from porcine stomach type II, SIGMA-ALDRICH) and 99.599 g of water (Milli Q). The solution was stirred for 30 min at ambient temperature to allow complete dissolution of mucin, and pH was adjusted to 7 with 35% NaOH (VWR, PROLABO). The artificial saliva was prepared every day and stored at 4 °C in-between experimental use.

2.4. Model bolus formation protocol

The evolution of the bolus was investigated by changing the saliva/cheese ratio. Five ratios were chosen in order to cover the range of