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Caramel odor: Contribution of volatile compounds according to their odor qualities to caramel typicality

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ABSTRACT

Odor of food constitutes one of the main drivers of the consumers' acceptance. Its characterization thus represents a main challenge for the food industry. Gas chromatography/olfactometry (GC/O) constitutes an intersection between physico-chemical and sensory studies by using the human nose as a detector to evaluate the odor properties of volatile compounds. As GC/O does not make possible the evaluation of mixture of odorants, we propose an original approach to evaluate the impact of compounds on the typicality of caramel aroma by considering their odor qualities in mixtures. Indeed, the present study relies on the main hypothesis that the qualitative properties are as important as quantitative one with regard to the typicality.

First, previously identified odorant compounds were distributed into eight odor categories using a classification wheel established from our GC/O descriptors. Each category was reconstituted separately and then a Whole Mixture was obtained by mixing all categories in specific proportions and validated by sensory analyses. Second, the impact of specific odor notes on the caramel typicality was studied individually by omission and addition tests and a 2^4 factorial design was built to investigate their interactions in complex mixtures.

The caramel typicality results from a complex balance between fruity, vegetal, sharp, nutty and caramel notes arising from the presence of carboxylic acids, aldehydes, oxygenated heterocyclic compounds, ketones and carbocyclic compounds. This study brings new clues to understand the contribution of the caramel volatile compounds to its odor while proposing a promising experimental approach to understand the contribution of volatile compounds to the odor of complex products.

1. Introduction

Odor of food constitutes one of the main drivers of the consumers' acceptance. For the last decades, advances in analytical sciences have improved our knowledge of food odors. However, the chemical composition is not sufficient to explain the odor perception (Piggott, 1990). Gas chromatography/olfactometry (GC/O) developed by Fuller, Steltenkamp, and Tisserand (1964) constitutes an intersection between physico-chemical and sensory studies by using the human nose as a detector to evaluate the odor properties of volatile compounds. If this technique has widely proved its efficiency to characterize individual odorant compounds in food products, it does not make it possible to study their contribution in mixture (Blank, 1997; d'Acampora Zellner, Dugo, Dugo, & Mondello, 2008; Delahunty, Eyres, & Dufour, 2006; Thomsen, Martin, Mercier, Tournayre, Berdague, Thomas-Danguin et al., 2012). Indeed, the existence of perceptual interactions occurring among odorants evidences the need to consider them in mixture to evaluate their contribution (Laing, 1994). In complex matrix as wine, Atanasova, Thomas-Danguin, Langlois, Nicklaus, Chabanet and Etievant (2005) showed that the addition of a woody odorant, even at low concentration, induced a significant change in the fruity odor, highlighting the existence of perceptive interactions between fruity and woody notes.

Many studies have attempted to develop methodologies based on aroma recombination. Some "key aroma compounds" are mixed to create an aroma model as close as possible to the original product. From this model, subsequent experiments are performed involving respectively omitting or adding by one a sub-set of compounds (Grosch, 2001). However, the main limitation concerns the selection of the "key odorant compounds". The most common methodologies are based on the idea that the higher the intensity, the higher the contribution to the whole aroma. Intensities are generally assessed through quantitative approaches such as Odor Activity Values (OAV) or dilution techniques such as Aroma Extract Dilution Analysis (AEDA) or Charm™ analysis (Acree, Barnard, & Cunningham, 1984). The OAV is the ratio of

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the concentration in the actual product to the detection threshold (Guadagni, Buttery, & Harris, 1966; Rothe & Thomas, 1963). In GC/O dilution techniques, OAVs are roughly estimated by a dilution value allowing establishing a hierarchical classification of the compounds. These techniques have shown relevant results giving an overview of some potent odorants for the aroma of specific food products (Callejon, Morales, Troncoso, & Ferreira, 2008; Ferreira, Ortín, Escudero, López, & Cacho, 2002). However, they also involve a simplistic outlook of the contribution of compounds to the aroma because they only take into account the detection threshold and not the whole relation existing between odor intensity and concentration given by psychophysical laws (Frijters, 1978). The Stevens' exponential law has been found as a good estimation of the perceived intensity in function of concentration (Stevens, 1960). Because the Stevens' slope is specific to each compound (Devos, Rouault, & Laffort, 2002), the OAVs, which do not take this parameter into account, are not a good estimate of the odor intensity at concentrations found in the original product. Moreover, the significance of sub and peri-threshold odorants in odor perception has been widely documented (Guadagni, Buttery, Okano, & Burr, 1963; Miyazawa, Gallagher, Preti, & Wise, 2008; Ryan, Prenzler, Saliba, & Scollary, 2008). In wine, Escudero, Gogorza, Melus, Ortin, Cacho and Ferreira (2004) studied the impact of the addition of two mercaptans with low OAV. It appeared that both of them were marked contributors to the wine aroma with the apparition of fruity and fresh notes that render the model much closer to the original wine. They concluded that despite low intensities those compounds are able to impact the aroma through the specificity of their aromatic notes. Based on the same assumptions, Campo, Saenz-Navajas, Herrero, Fernandez-Zurbano, and Ferreira (2013) constructed a wine model from a common base supplemented by six target odor families. They showed that the impact of a single family is highly conditioned by the blend context. Following this idea, we developed an original approach based on odor qualities of the volatile compounds to evaluate their impact on caramel odor typicality.

In many studies, recombined models are evaluated with quantitative descriptive analyses (Callejon et al., 2008; Pang, Chen, Hu, Zhang, & Wu, 2012). Consequently, the impact of key compounds or odor notes on the whole odor was not directly evaluated but only estimated by their impact on sensory attributes potentially important for the odor product. For example, in a study on red wine, authors focused on berry-fruit, sweet-caramel, toasty and phenolic attributes known to be linked to the wine typicality (Escudero, Campo, Farina, Cacho, & Ferreira, 2007). In 1994, Dacremont & Vickers assessed the importance of volatile compounds for Cheddar cheese aroma by the concept matching technique (Dacremont & Vickers, 1994). It involves the classification of the models according to their closeness to the Cheddar cheese concept. Another original approach was proposed for studying the Chardonnay wine odor with three groups of compounds considered as positive, negative or neutral contributors (Jaffré, 2009). By supplementing wine with those groups and evaluating the Chardonnay typicality of the resulting mixtures, the authors expected to confirm their predicted contribution. As an alternative to the description of mixtures by descriptive approaches, the assessment of typicality appeared to be an efficient way to evaluate the contribution of the odorants to the whole product. Although, we recently attempted to explain odor properties of four different caramels with GC/O data by means of a multivariate statistical technique (Paravisini, Gourrat-Pernin, Gouttefangeas, Moretton, Nigay, Dacremont et al., 2012), the direct contribution of odor notes to caramel odor has not been studied yet. The present study relies on the main hypothesis that the qualitative properties of odorant compounds are as important as quantitative one and have to be considered. From the GC/O and GC/MS results of our previous study, identified odorant compounds were distributed into eight odor categories according to their odor quality. For each category, compounds sharing similar odor qualities were mixed up to achieve one mixture representative of the odor note of this category. Then, the impact of those eight odor notes on the caramel typicality was studied individually by omission and addition tests. Moreover, taking into account interactions between odorants in mixture requires the use of a specific experimental design. The most suitable methodologies involve the use of factorial designs. Hallier et al. proposed a fractionated factorial design to evaluate the impact of five odor families by omission tests, allowing the estimation of main effects and first order interactions (Hallier, Courcoux, Serot, & Prost, 2004). However, as we hypothesize that the caramel odor is the result of complex interactions between odor notes, it implies the use of a full factorial design to evaluate high-order interactions as well. Thus, a 2^4 factorial design was built to study the interactions among the four more relevant odor notes in mixtures.

2. Materials and methods

2.1. Standards

All the standards used were purchased from Sigma-Aldrich (Saint Quentin-Fallavier, France) in food grade and with the highest purity available (Table 1). Compounds were diluted in Milli-Q water and anhydrous ethanol (>99%, pharmaceutical grade) purchased from Carlo-Erba (Val de Reuil, France).

The sugar syrup and the caramel samples were provided by Nigay S.A.S. (Feurs, France).

2.2. Recombination of caramel aroma

The recombination was led in 3 steps: 1) classification of the volatile odorant compounds into 8 odor categories; 2) formulation of 8 odorant blends (B) representative of each odor category and 3) formulation of a Whole Mixture (WM) by mixing up the 8 blends.

2.2.1. Classification of the odor notes

In a previous study led on four caramel samples, 76 odorant zones (OZs) were detected by GC/O analyses performed with nine panelists in duplicate (Paravisini et al., 2012). Each odorant zone (OZ) was associated with a detection frequency, a list of descriptors generated by the panel and the corresponding odorant compound whenever identified by GC/MS. In a first step, the descriptors were organized into a classification wheel. As the odors cannot be linked to physicochemical features of compounds (Kaeppler & Mueller, 2013), they are usually classified according to the meaning of their descriptors. As there is no universal classification tool, an ad hoc classification wheel was developed for our data. First, hedonic and intensity terms were eliminated. Synonyms were grouped based on their semantic meaning. Then, the remaining 300 terms were organized into 40 subcategories finally grouped into eight main categories: caramel, roasted, fruity, vegetal, floral, nutty, animal and sharp (Fig. 1). Some attributes did not belong to one of those categories and were put in a miscellaneous category. This last category was not considered as relevant for the odor quality and thus was not taken into consideration in subsequent experiments.

For each OZ, the most often cited term was selected as representative of the quality of this OZ and the OZ was assigned to one odor category among the eight using the classification wheel.

2.2.2. Formulation of the odorant blends (B)

The 21 OZs for which one compound was positively identified and was commercially available at safety grade, were considered. These odorants were used to prepare the eight odorant blends, one by odor category. Concentrations were chosen on the basis of quantification. Quantification was done by standard addition; pure compounds (Table 1) were added at three concentration levels (in triplicate) in aromatic caramel and calibration curves were established according to gas chromatography/mass spectrometry data. The concentrations in odorant blends were adjusted according to sensory data to obtain the best compromise between iso-intensity across odorant blends and odor quality representative of each category.