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Combining untargeted, targeted and sensory data to investigate the impact of storage on food volatiles: A case study on strawberry juice

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ABSTRACT

An integrated science-based approach, combining analytical and sensorial data and different data analysis methods, proved successful to study the impact of storage time, storage temperature and oxygen availability on strawberry juice volatiles and allowed to get a multi-perspective view on these changes. An untargeted GC–MS approach showed that the volatile fraction of shelf-stable strawberry juice clearly changed during ambient storage and that oxygen availability (linked to the type of bottle) had a limited effect. To gain further insight, several characteristic aroma compounds were quantified during storage at ambient (20 °C) and accelerated (28–42 °C) temperatures, kinetic parameters were estimated and odour activity values were calculated. The kinetic parameters showed that all characteristic aroma compounds changed significantly during storage at all temperatures and that the rate of change in some compounds was accelerated by storage at higher temperatures. The observed changes in strawberry juice volatiles caused sensorial differences between non-stored and 20 °C stored samples as shown by the sensory evaluations and odour activity values.

1. Introduction

The analysis of food products is a research area which is continuously improving. These advances are partially driven by consumer demands concerning food safety and quality issues, the diversity of food products available on the market (including new products) and the need for accurate shelf-life predictions (Cevallos-Cevallos, Reyes-De-Corcuera, Etxeberria, Danyluk, & Rodrick, 2009; Ibáñez, Simó, García-Cañas, Acunha, & Cifuentes, 2015; Nicoli, 2012). Nevertheless, it should be noted that food quality investigations and shelf-life assessment are complex. This can be addressed to the fact that food quality is defined as the sum of all different properties and assessable attributes of a food product (e.g. colour, flavour, texture, vitamin content) (Leitzmann, 1993). These attributes are affected by different intrinsic (e.g. composition, structure) and extrinsic factors (e.g. storage conditions, oxygen availability), thereby making the study of quality changes of food products even more complicated (Kilcast & Subramaniam, 2000).

In the past and still today, many researchers successfully applied a targeted approach to study quality changes during processing and storage. A traditional targeted approach focusses on specific and known compounds or reactions. As food products are complex systems in

Abbreviations: ASLT, Accelerated shelf-life testing; HS-SPME-GC-MS, Headspace Solid Phase Micro Extraction Gas Chromatography Mass Spectrometry; LV(s), Latent variable(s); MPP, Mass Profiler Professional; MVDA, Multivariate data analysis; OAV, Odour activity value; OTR, Oxygen transfer rate; PLS, Partial least squares; RI, Retention index; VID, Variable Identification

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which multiple reactions are occurring simultaneously, some of these reactions can be of minor importance, while other important but unknown reactions maybe overlooked. Therefore, the application of an untargeted multivariate approach has been suggested and has proven to be useful to study food quality changes (Castro-Puyana, Pérez-Míguez, Montero, & Herrero, 2017; Grauwet, Vervoort, Colle, Van Loey, & Hendrickx, 2014; Kebede et al., 2015a, 2015b). In such an approach, no compounds are selected beforehand, thereby aiming to detect as many chemical compounds as possible in the food product under investigation and opening the opportunity to detect unexpected and unknown reactions.

Besides studying quality changes using analytical approaches, the interaction of the food product with consumers will determine whether it is appreciated. In other words, performing standardised sensory analyses is becoming more and more important in a scientific approach to study food quality and should not be neglected. In a final step, the analytical and sensorial data can be integrated to get a multi-perspective view on quality changes.

Besides analytical progress, the potential of a wide range of statistical data analysis techniques for food quality investigations is not yet fully exploited. For example, chemometric tools are very useful to detect changes in untargeted multivariate data and kinetic modelling allows to describe quality changes quantitatively by estimating kinetic parameters (e.g. reaction rate constants) (Castro-Puyana et al., 2017; Cevallos-Cevallos et al., 2009; van Boekel, 2008). An increase in the use of such data analysis methods can result in a further progression in the research area of food and in a better understanding of quality changes.

Taking into account the complexity of food products, Wibowo and co-workers recently presented an integrated science-based approach for quality investigations during storage and shelf-life assessment. In such an approach it is suggested to obtain data by using different analytical methods (e.g. untargeted, targeted), to apply different types of data analysis (e.g. chemometrics, kinetic modelling) and to include sensory evaluations (e.g. difference testing, acceptance testing) (Wibowo, Buvé, Hendrickx, Van Loey, & Grauwet, 2018). To show the potential of the aforementioned approach, present work applied an integrated science-based approach to study volatile changes during storage using untargeted, targeted and sensory analyses next to the use of advanced data analysis techniques. Strawberry juice was selected as a case study. In this work, attempts were made to answer following research questions using the integrated science-based approach: (i) Is the volatile fraction of shelf-stable strawberry juice changing during ambient storage? (ii) Are the human senses sensitive to volatile changes during ambient storage? (iii) Which aroma compounds are causing sensorial differences? (iv) Does oxygen availability and storage temperature have an effect on volatile changes during storage?

2. Material and methods

2.1. Preparation and storage of shelf-stable strawberry juice

The applied processing steps were extensively described in our previous work (Buvé, et al., 2018a, b). Preparation of shelf-stable juice was performed at Food Pilot (Melle, Belgium) under food grade conditions. Fresh strawberries (*Fragaria x ananassa*, cv. Elstanta) were shredded under an inert nitrogen atmosphere. A spiral-filter press (VaculIQ 1000–300, VaculIQ GmbH & Co. KG, Hamminkeln, Germany) was used to extract strawberry juice from the shredded biomass under vacuum conditions. The fresh juice was pasteurised (2 min, 95 °C) in a tubular heat exchanger (APV SPP, SPX Corporation, Gatwick, UK) to obtain a shelf-stable juice. The pasteurised juice was filled under aseptic conditions into two types of PET bottles (500 ml) with a different OTR (oxygen transfer rate): a standard monolayer bottle and a multilayer bottle with a reduced OTR. The bottles were closed by induction sealing with a screw cap and aluminum liner. Closed mono- and multilayer bottles had an OTR of 0.0437 and 0.0049 cm^3 per bottle per day at 20 °C, respectively. Bottles and caps were supplied by Resilux NV (Wetteren, Belgium) and were sterilised beforehand by gamma radiation. Filled monolayer bottles were stored at 20 °C (32 weeks), 28 °C (32 weeks), 35 °C (12 weeks) and 42 °C (8 weeks) in incubators protected from light. Filled multilayer bottles were stored at 20 °C. Sampling was performed 12 times during storage. At each sampling time, bottles were collected randomly from an incubator and samples were stored at −40 °C until analysis. Prior to sample preparation, samples were thawed overnight in a cooling room (4 °C).

2.2. Untargeted and targeted HS-SPME-GC–MS approach

Volatile changes were analytically studied at two levels: (i) by an untargeted semi-quantitative GC–MS approach and (ii) by a targeted quantitative GC–MS approach. The untargeted approach resulted in a volatile profile of the strawberry juice. The targeted approach focussed on twelve important and characteristic aroma compounds of strawberry juice: ethyl, butyl and hexyl acetate, methyl and ethyl butanoate, ethyl hexanoate, linalool, α-terpineol, 2-hexenal, hexanal, acetic acid and dimethyl sulfide. These compounds were selected based the outcome of the untargeted approach and their importance for strawberry aroma (Pérez & Sanz, 2010). All characteristic aroma compounds could be detected in one analysis.

All analyses were performed on a gas chromatography system (7890A, Agilent Technologies, Diegem, Belgium) connected to a mass selective detector (5975C, Agilent Technologies, Diegem, Belgium) and equipped with a CombiPal autosampler (CTC analytics, Zwingen, Switzerland) with a cooling tray (set at 10 °C).

2.2.1. Sample preparation for untargeted analyses

Samples stored at 20 °C in both bottle types were analysed using an untargeted approach. Strawberry juice and saturated NaCl solution were transferred to a 10 ml dark glass vial (1:1 w/v ratio, 1.5 g of juice and 1.5 ml of saturated NaCl solution). Vials were closed with metal screw caps with a silicon septum. For each storage time, six replications were prepared which were analysed once. This resulted in six volatile profiles per storage time for each bottle type.

2.2.2. Sample preparation for targeted analyses

Aroma compounds were quantified for the samples stored at all four temperatures in the monolayer bottle. Sample preparation steps are comparable with the one of the untargeted analyses (Section 2.2.1), however due to some small differences, the different steps were described. Thawed strawberry juice (1.5 g) was transferred into a dark vial (10 ml) together with demineralised water (900 µl) and saturated NaCl solution (1.5 ml). Vials were closed with metal screw caps with a silicon septum. 100 µl internal standard (diluted 3-heptanone solution) was added to each vial with a gastight syringe (Du, Plotto, Baldwin, & Rouseff, 2011). Vials were weighed after each step to know the exact amount of the added solutions. Sample preparation was performed in triplicate for each storage condition (i.e. combination of storage time and storage temperature).

2.2.3. Headspace-SPME-GC–MS method

The same HS-SPME-GC–MS method could be used for the untargeted and targeted approach. This method was based on the one described by Kebede et al. (2015a) and was optimised beforehand. Both GC–MS analyses were performed independently. Samples were incubated for 20 min at 40 °C in the incubation oven of the autosampler under agitation (500 rpm). Next, a 30/50 µm DVB/CAR/PDMS fiber (StableFlex, Supelco, Bellefonte, Pennsylvania, USA) was exposed to the headspace of the vial for 10 min at 40 °C under agitation. The adsorbed volatiles were thermally desorbed from the SPME fiber at the GC injection port (2 min at 230 °C). Injection was performed in split mode (1/5). After injection, the fiber was thermally regenerated (270 °C for 5 min). Volatile compounds were separated on a capillary HP