Food Research International 45 (2012) 17–25

Contents lists available at SciVerse ScienceDirect

Food Research International

journal homepage: www.elsevier.com/locate/foodres

Flavour profiling of fresh and processed fruit smoothies by instrumental and sensory analysis

Derek F. Keenan [a,*], Nigel P. Brunton [a], Michelle Mitchell [a], Ronan Gormley [b], Francis Butler [c]

[a] Teagasc, Food Research Centre, Ashtown, Dublin 15, Ireland
[b] UCD Institute of Food and Health, University College Dublin, Dublin 4, Ireland
[c] Biosystems Engineering, UCD School of Agriculture, Food Science and Veterinary Medicine, University College Dublin, Dublin 4, Ireland

ARTICLE INFO

Article history:
Received 28 April 2011
Accepted 3 October 2011

Keywords:
Smoothies
HHP
Sensory profiling
Flavour volatiles
SPME
GC–MS

ABSTRACT

Flavour volatile compounds and descriptive sensory attributes were examined in fresh, thermal and high hydrostatic pressure (HHP) processed (450 MPa/5 min/20 °C or 600 MPa/10 min/20 °C) fruit smoothies stored over 30 days (4 °C). Volatile compounds were extracted by solid-phase microextraction (SPME) and analysed by gas chromatography mass spectrometry (GCMS). Limonene and trans-2-hexanal were the most abundant compounds present in the smoothies. Processing affected the limonene content of smoothies, with thermally processed samples shown to be higher ($p<0.05$) than all other samples. As expected, storage decreased ($p<0.05$) the volatile content of all smoothies, in particular for volatiles associated with 'fresh/green/grassy' flavour notes, like trans-2-hexanal. Principal component analysis (PCA) of descriptive sensory data revealed that attributes such as pink colour, fresh colour, apple aroma, fresh aroma, and fresh flavour were more associated with both fresh and processed smoothies on day 0. Physico-chemical assessment showed that colour was affected ($p<0.05$) by both processing and storage but the changes were not consistent for the individual processing types i.e. HHP treated samples showed good initial colour retention but had the biggest overall changes over time, while thermal treatments initially degraded colour but had the lowest colour change over time. Partial least squares (PLS) regression was used to investigate the relationships between sensory attributes and volatile compounds. However, correlations between sensory and instrumental data were lower than expectations, possibly due to the high odour threshold values of the compounds identified.

1. Introduction

Smoothies are blended beverages containing fruit, fruit juice, ice, yoghurt, milk; and are a popular way of consuming fruit (Safefood, 2009). These products are typically purchased freshly prepared from juice bars or as a processed product (mildly pasteurised) from the chilled section of retail outlets. Despite worsening global economic conditions, smoothies remain a popular and convenient way of consuming fruit. In fact, the world smoothie market is projected to touch $9 billion by the year 2015 (Global Industry Analysts, 2010). This is primarily driven by rising health consciousness among consumers, on-the-go consumption, convenience, and perceived fresh-like taste offered by smoothies. While an ideal processed product would maintain all the sensory perceptions of a freshly prepared product, the chemical changes induced by processing and post-production alter the sensory profile of its specific components (Perez-Cacho & Rouseff, 2008). Aroma is key determinant of quality in fruits and this in turn is a function of the volatile profile of a foodstuff. Therefore, in single component foods much effort has been directed towards relating level of aroma volatiles to the sensory profile as determined using a trained panel. Sensory profiling, also known as descriptive analysis, is the process during which a panel of trained assessors score several sensory attributes of a product. It is the technique of choice for relating information of aroma volatiles to sensory perception as it gives detailed insights into panellist's perceptions of a number of flavour notes which can be related to levels of individual aroma volatiles. However, there is limited information on this relationship for multi-component foods such as fruit smoothies. This information is of value to processors as it facilitates an understanding of the influence of processing on the levels of key aroma volatiles and ultimately the sensory profile of the food. For example, thermal processing is undoubtedly the most common and cost effect method to extend the shelf-life by reducing microbial numbers and enzyme activity. However, this type of processing can reduce the concentration of volatiles commonly associated with fresh products and initiate reactions that result in the formation of off-flavours (Bazemore, Goodner, & Rouseff, 1999). Therefore, in pursuit of a techniques that can both extend shelf life but produce a fresh-like products in terms of flavour non-thermal pasteurisation processing methods are being examined. (Gui et al.,

* Corresponding author. Tel.: +353 18059505; fax: +353 18059550.
E-mail address: derek.keenan@teagasc.ie (D.F. Keenan).

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doi:10.1016/j.foodres.2011.10.002

2007; Heinz, Toepfl, & Knorr, 2003). High hydrostatic pressure (HHP) is one such technique and has been the subject of much research (Oey, Lille, Van Loey, & Hendrickx, 2008; Oey, Van der Plancken, Van Loey, & Hendrickx, 2008; Rastogi, Raghavarao, Balasubramaniam, Niranjan, & Knorr, 2007). Due to the stability of covalent bonds to high pressure (Knorr, 1993), HHP should in theory, have a reduced smaller molecules such as volatile compounds associated with the sensory quality (Cheftel, 1992). Research has indicated however that HHP can both enhance and diminish enzymatic and chemical reactions involved in the formation and degradation of aroma compounds (Oey, Lille, Van Loey, & Hendrickx, 2008). In fact, previous work carried out by this group reported that sensory panellists noted a decrease in aroma and flavour intensity in HHP processed (600 MPa/20 °C/15 min) smoothies after 28 days storage compared to fresh controls (Gormley et al., 2009). Therefore, a principal objective of the present study was to use the combined approach of sensory and instrumental volatile, colour and rheological analyses to better elucidate the effect of processing (HHP/thermal) on the overall sensory properties of fruit smoothies during chill storage for 30 days.

2. Material and methods

2.1. Chemicals

Butylacetate, hexanal, amylacetate, 3-carene, limonene, trans-2-hexanal and hexylacetate were obtained from Sigma Aldrich (Dublin, Ireland).

2.2. Sample preparation

Smoothie products were prepared as previously described by Keenan, Brunton, Gormley, and Butler (2011). Strawberries (*cv.* Sabrosa; Spain), apples (*cv.* Braeburn; France), apple juice from concentrate (Tesco value, Ireland), bananas (*cv.* Nino; Cameroon) and oranges (*cv.* Navel-late; Spain) were obtained from a local retailer. Smoothie composition by weight was whole apple (29.5), apple juice from concentrate (29.5), strawberry (21), banana (12) and orange (8%). Fruit was blended in a homogeniser (Robot Coupé Blixer 4® mono, Bourgogne, France) for 3 min. All smoothie samples were filled to an equal height and sealed into 250 ml HHP grade polyethylene terephthalate bottles (The Packaging Centre, product code 18PBC250J, Dublin, Ireland). Controls (fresh) were chilled (2–4 °C) immediately, while other samples were subjected to subsequent processing.

2.3. High hydrostatic pressure (HHP) and thermal processing treatments

HHP processing was carried out as previously described by Keenan et al. (2011) in a high pressure vessel (100 mm internal diameter × 254 mm internal height, Pressure Engineered System, Belgium). Samples were pressurized at 600 and 450 MPa for 10 and 5 min respectively, at 20 °C ('H450' and 'H600'). Temperature within the pressure chamber increased from 20 to 37.5 °C during high pressure processing. During this time, sample temperature increased by an average of 2 °C. A mild thermal pasteurisation was applied to ensure adequate shelf-life stability and maximum retention of the volatiles. For thermal processing, calibration of temperature probes and core temperature profiles of fruit smoothies were recorded as described by Keenan et al., 2011. Samples were loaded into a pilot scale retort (Barriquand Steriflow, Roanne, France) and pasteurised ($P_{70} \geq 10$ min) (Gould, 1999). All processed and fresh samples were stored for 0, 15 and 30 days at 4 °C.

2.4. Chemical and physical analysis

Headspace volatile of smoothies in bottles were collected and concentrated using solid phase microextraction (SPME) and analysed using GC–MS. Prior to SPME analysis, a series of experiments was conducted to determine the optimal sampling conditions in terms of reproducibility and reducing sampling time. The conditions described below represent the best compromise between reduced sampling time and optimal reproducibility. 10 ml of sample was pipetted into a bottle/vial (25 ml) containing a magnetic stirrer. Samples were equilibrated for 5 min at 40 °C in a thermostatically controlled water bath followed by exposure of an 85 μm carboxen-polydimethylsiloxane (CAR-PDMS) SPME fibre (Supelco, Bellefonte, PA, USA) to the headspace of each vial for 30 min. The SPME needle was introduced to the headspace of the bottle via an adhesive septum applied to the bottle. Analysis of the volatile compounds adsorbed on the fibre was carried out using a Varian 3800 GC coupled to a Varian Saturn 2000 ion trap mass spectrometer (Varian Chromatography Systems, Walnut Creek, CA, USA). Separation of the volatiles was accomplished on a ZB-wax column (60 m × 0.25 mm i.d., 0.25 μm film, Phenomenex Inc, Torrance, CA, USA). Helium, at a flow rate of 1.2 ml min^{-1}, was used as the carrier gas. Thermal desorption of the compounds took place in the GC injection port (1079 Programmable Temperature Vaporizing (PTV) Injector), equipped with a 0.75 mm i.d. splitless glass liner, at 290 °C for 7 min in splitless mode. The split valve was then opened (ratio 1:100) for a duration of 3 min and then returned to splitless mode for a further 13.5 min. The fibre remained in the injection port for approximately 2 min. The oven temperature was programmed at an initial temperature of 45 °C for 4 min, increased to 150 °C at a rate of 10 °C min^{-1} and then further increased to a final temperature of 250 °C at a rate of 25 °C min^{-1} at which point the temperature remained constant for 5 min. The MS transfer line temperature was set to 260 °C. The mass spectrometer was tuned using the auto-tune procedure and masses from m/z 33 to 400 were recorded after electron impact ionisation under EI auto mode. Peak areas were analysed and quantified using the Varian star chromatography workstation software (v 5.0; Varian Chromatography Systems). Compounds were identified by the use of authenticated standards (Sigma-Aldrich Corporation, St. Louis, MO, USA) and by matching mass spectra with the data stored in the NIST library of standard compounds.

2.5. Sensory profiling analysis

Sensory profiling analysis (flavour profile method) was conducted on the fresh and processed (thermal or HHP) smoothies as previously described by Mitchell, Brunton, and Wilkinson (2011). A panel of twelve members, 5 male and 7 females in their twenties and thirties, was selected and trained according to the guidelines set out in ISO 8586–1: 1993(E) and in ISO 6564:1985(E). The panellists' were selected from staff and students of the Teagasc, Food Research Centre, Ashtown, Dublin, all of whom had previous sensory experience. Screening using a series of acuity and discrimination tests (ISO 8586–1: 1993(E)) was conducted and panellists were required to correctly complete all sessions to be eligible for inclusion in the study. The selected panel members were trained and a set of descriptors was developed and defined for smoothie samples during training, which encompassed appearance, aroma, flavour and textural attributes, order of perception, intensity and overall acceptability. A total of 17 descriptors were developed and agreed upon by panel consensus (Table 1). Sensory profiling was carried out in a sensory laboratory with individual testing booths equipped with serving windows and controlled lighting. The panellists were presented with a total of 4 samples per session using a completely randomized sample presentation order to minimise bias due to first-order and carry-over effects (Baxter, Easton, Schneebeli, & Whitfield, 2005; MacFie, Bratchell, Greenhoff, & Vallis, 1989). Intensity ratings for each of the descriptive terms were scored using a 15 cm line scale ranging from low intensity (0 cm) corresponding to the word anchor 'none' to high intensity (15 cm) corresponding to the word anchor 'strong'. In a pre-test