



Influence of the production process of strawberry industrial purees on free and glycosidically bound aroma compounds

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ABSTRACT

A portion of the odourless fraction of fruits bound to sugars releases aromatic substances that represent an important source of aromatic potential. During the processing of fruits, these compounds may be affected. Thus, in this work, for the first time, glycosidic aroma precursors were studied over the course of the industrial production process of commercial strawberry puree. Free volatile compounds were also studied. The results indicated that the amounts of free and bound aromatic compounds decreased, particularly in the free fraction, by more than 50% of the total amounts. The pasteurisation stage led to the greatest loss of the precursors of key strawberry odorants. However, the seed removal step offset these losses during the processing of glycosidically bound aroma compounds. The free volatile compounds that were most strongly affected were the higher alcohols and ethyl esters. This study suggests that the amounts of glycosidic aroma precursors in the raw material significantly affect the aromatic potentials of commercial purees.

Industrial relevance: In producing country strawberries, every year, part of this crop is discarded due to several reasons like size, deformations or even overproduction, which cause surpluses.

These strawberries of second quality are suitable for human consumption. So, these are used to obtain different products such as purees. These purees are provided to other industries that use it as an ingredient in the production of fruit-based commodities. In these products the aroma and aromatic potential which is due to the content of odourless aroma precursors are important. So, to conserve and to enhance aromatic potential are necessary to know how the industrial production process of commercial strawberry puree affects aroma precursors and volatile compounds. To the best of our knowledge, the effect of fruit processing on these odourless aroma precursors has not yet been studied. We consider that the results of this study are relevant to improve the product quality and economic benefits in the industry that develops products from strawberry.

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1. Introduction

Currently, products obtained from processed fruits provide alternative uses for fruit surpluses. Notably, 25% of all strawberries produced are employed for this purpose. Such fruit processing may comprise several treatments, including heating, filtration, enzymation, mechanical compression, air-drying, rehydration and ultra-high pressure (Butz et al., 2003; Falade & Abbo, 2007; Jaeger de Carvalho, Miranda de Castro, & Bento da Silva, 2008; Zhou, Xu, Sun, & Wang, 2002). These processes result in changes in the nutritional and organoleptic properties of the processed materials (Oeya, Lilleb, Loeya, & Hendrickx, 2008; Pérez, Luaces, Oliva, Rios, & Sanz, 2005).

Several industries specialise in the processing of fruit to obtain purees. These purees are provided to other industries that use it as an ingredient in the production of fruit-based commodities. To obtain

puree, fruit is submitted to different processes such as mashing, enzymatic inactivation, pasteurisation, etc.

Aroma is one of the most valued attributes of fruit and one of the characteristics that changes during the processing.

Fruit aromas are composed by large numbers of volatile compounds (Berger, 2007; Brückner, 2008). Moreover, fruits contain non-volatile compounds called aroma precursors that are present mainly as glycoconjugates formed by a sugar and an aglycone. These compounds are a potential natural source of aroma because hydrolysis of the sugar-aglycone bond turns these molecules into aromatic compounds. Aglycones of strawberry aroma precursors are mainly furanones, lactones, terpenes and benzenes among others (Ubeda et al., 2012a). The importance of these compounds is that many of these compounds are impact odorants of strawberry (Mayerl, Naf, & Thomas, 1989; Pérez, Olías, Sanz, & Olías, 1996).

During ripening, the bond between the sugar and the aglycone is broken, releasing the strawberry aroma (Groyné, Lognay, & Marlier, 1999). However, to the best of our knowledge, the effect of this fruit processing on these aroma precursors has not yet been studied.

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The industrial process to obtain derivatives products from strawberry should try to preserve the highest content in compounds involved in the aroma of this fruit (free and bound volatiles).

Thus, the aim of this work was to evaluate the influences of each stage of the industrial processing of strawberries to obtain commercial puree on aroma precursor glycoconjugates. Moreover, analyses of free volatile compounds were also performed.

2. Materials and methods

2.1. Samples

To study the influence of the different treatments of the industrial production of strawberry purees from fresh fruit (*Fragaria* × *ananassa* Duch. var 'Camarosa'), two production processes were studied over the harvests of 2011 (11) and 2012 (12). These purees were purchased from Hudisa company (Huelva, Spain). Two samples were taken at each step of the production process and were stored at -20°C until the analysis. The first sample of each process was a strawberry mash with seeds (MS). The second sample (EIS) was taken after enzymatic inactivation, which consisted of subjecting the MS to $55\text{--}65^{\circ}\text{C}$ for 2 min. Next, then after this, the mashes were filtered through a sieve with 1.5 mm pores and pasteurised at 90°C for 180 s to obtain purees with seeds (PWS). A portion of this product is commercially available without seeds, and to obtain this puree free of seeds (PFS), the PWS was passed through a sieve with 0.5 mm pores.

2.2. Extraction and analysis of glycoside aroma precursors

To extract the aroma precursors, a 10 g sample was spiked with 100 μL of an ascorbic acid solution (50 mg/L) to prevent oxidation and was then centrifuged for 10 min at 4000 rpm. Glycoside precursor extract was obtained following the method described by Ubeda et al. (2012a). The extract was reconstituted with 10 mL of citric buffer (0.2 M, pH 2.5), and acid hydrolysis was carried at 100°C over 1 h. Each extraction and hydrolysis was performed in duplicate. After hydrolysis, the released volatile compounds were recovered following the procedure of Ubeda et al. (2012a).

The obtained extracts were concentrated to 100 μL with a gentle nitrogen stream and spiked with 5 μL of the internal standard (IS) 4-methyl-2-pentanol (405 mg/L).

2.3. Liquid–liquid extraction of volatile compound free

The liquid–liquid extraction method was utilised to obtain representative extracts of the samples. This procedure was based on that of Callejón, Morales, Troncoso, and Silva-Ferreira (2008) with the following modifications: 2.5 g of anhydrous sodium sulphate was added to 25 mL of each sample and was extracted twice over 5 min with 5 mL of dichloromethane using a magnetic stir bar. Next, 4 mL of the organic phase was concentrated 8 times under vacuum, and 200 μL of the extract was spiked with 10 μL of IS 4-methyl-2-pentanol (405 mg/L). The extraction of all samples was performed in duplicate.

2.4. GC–MS analytical conditions

Analyses were conducted using an Agilent 6890 GC system coupled to an Agilent 5975 inert quadrupole mass spectrometer equipped with a Gerstel MP2 autosampler (Müllheim an der Ruhr, Germany). A DB-WAX capillary column (60 m × 0.25 mm × 0.32 μm film thickness; J & W Scientific, Agilent Technologies Inc., Santa Clara, USA) was employed. The carrier gas was helium.

For aroma precursor analyses, the oven temperature was programmed as follows: 3 min at 40°C , increased by $4^{\circ}\text{C}/\text{min}$ to 80°C , increased by $2^{\circ}\text{C}/\text{min}$ to 230°C , and 230°C for 37 min. Four microlitres of the sample was injected in splitless mode, and the injector temperature was 230°C . The flow rate of the carrier gas was 1 mL/min.

For analyses of the free volatile compounds, the oven temperature was programmed as follows: 40°C for 1 min, increased by $2^{\circ}\text{C}/\text{min}$ to 220°C , and 220°C for 40 min. Four microlitres of sample were injected in splitless mode, and the injector temperature was 220°C . The flow rate of the carrier gas was 1.3 mL/min.

In both cases, the quadrupole, source and transfer line temperatures were maintained at 150, 230 and 280°C , respectively. Electron ionisation mass spectra data from m/z 29–350 for the free volatile compounds and 40–220 for the precursor were collected in scan mode with an ionisation voltage of 70 eV. All data were recorded using an MS ChemStation. The samples were analysed in duplicate, and blank runs were performed prior to and following each analysis.

2.5. Qualification and quantification

The volatile compounds were identified based on comparisons of the linear retention indices (LRIs) of the standards and computer matching to the reference mass spectra from the NIST 98 library. When standards were not available, the compounds were identified by computer matching to the reference mass spectra from the NIST 98 library and by the comparison of their LRIs with the LRIs obtained with standards that have been reported in the literature. The remaining compounds were tentatively identified by computer matching to the reference mass spectra from the NIST 98 library and/or through comparisons of their LRIs with those of online databases (Flavornet; Pherobase) and the literature. LRIs were calculated based on the retention times of the n-alkanes (C10–C32) under conditions that were identical to those of the analyses.

The quantitative determination of volatile compounds was performed using the relative area, which was calculated as the ratio of the target ion of each compound and the IS. Calibration curves were built by injecting seven concentration levels and three replicates per level. The range of the calibration curves was chosen to cover the possible concentrations of the real samples.

2.6. Statistical analyses

Analysis of variance (ANOVA) and principal component analysis (PCA) were performed using the Statistica (version 7.0) software package (Statsoft, Tulsa, USA).

Notes to Table 1

TI: compounds tentatively identified when mass spectrum agreed with mass spectral data base or LRI agreed with literature data.

n.q: concentration under quantification limits.

n.d: concentration under detection limits.

^a Significant differences with previous stage.

^b Significant differences with initial mash.

^c Values of relatives areas.

^d Identification based on mass spectrometry data and the coincidence of experimental LRI with literature LRI obtained with standards:

^e(Culleré, Escudero, Cacho, & Ferreira, 2004),

^f(Högnadóttir & Rouseff, 2003).

^g Relative areas not included for the total sum.

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