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## Influence of storage on the volatile profile, mechanical, optical properties and antioxidant activity of strawberry spreads made with isomaltulose

Irene Peinado<sup>a,\*</sup>, Estela Rosa<sup>b</sup>, Ana Heredia<sup>b</sup>, Isabel Escriche<sup>b</sup>, Ana Andrés<sup>b</sup>

<sup>a</sup> Faculty of Science and Technology, Free University of Bolzano, Piazza Università 5, 39100 BZ, Italy
<sup>b</sup> Institute of Food Engineering for Development, Universitat Politècnica de València, P.O. Box 46022, Valencia, Spain

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Chemical compounds studied in this article: Isomaltulose (PubChem CID: 439559) Sucrose (PubChem CID: 5988) Fructose (PubChem CID: 5984) Citric acid (PubChem CID: 311) Potassium sorbate (PubChem CID: 23676745) 1,1-Diphenyl-2-picrylhydrazyl radical (DPPH) (PubChem CID: 2735032) isopropyl acetate (PubChem CID: 7915) Propan-2-ol (PubChem CID 3776) 2-buten-1-ol (PubChem CID 20024)

1. Introduction

Strawberry spread-product refers to a spread sweet food with improved functionality due to the replacement of sucrose by healthier sugars, such as isomaltulose, and to the incorporation of a higher content of fruit compared to jams for instance. The usual cooking step of jam processing to reach a final soluble solid concentration is avoided in fruit spread-product manufacturing. In our previous study, the influence of processing parameters such as, fruit, sugars (isomaltulose–sucrose or isomaltulose–fructose), pectin and citric acid, on the volatile profile of strawberry spread-product formulated with isomaltulose and sucrose or fructose, was analysed (Peinado, Rosa, Heredia, Escriche & Andrés, 2013). A major effect of

\* Corresponding author.

*E-mail addresses:* Irene.PeinadoPardo@unibz.it, irpeipar@gmail.com (I. Peinado).

#### ABSTRACT

This work represents the final step of a series of studies on the formulation of strawberry products with partial replacement of sucrose by healthier sugars such as fructose and isomaltulose. Previously, quality parameters of the formulated products such as colour, texture, rheology, aromatic profile and sensory evaluation were assessed. As a final step, in the present work, the volatile profile evolution of a strawberry spread-product during 90 days of storage at room temperature (20 °C), and its relation with some physicochemical properties ( $a_{w}$ , pH, texture and colour) and antioxidant activity as well as anthocyanin content were studied.

Most of the volatile compounds modified their concentration during storage; some of them totally disappeared but 13 new compounds were formed: (methyl-2-methyl butyrate, E-2-butenal, 2-butenal-2-methyl, 2-buten-1-ol, 2-penten-1-ol, 2-etil-1-hexanol, 6-methyl-5-hepten-2-one, acetic acid, propanoic-2-methyl acid, butyric acid and butyric-2-methyl acid). Storage led to a dark pink colour and an increase in consistency and adhesiveness while the antioxidant activity considerably increased (from  $18 \pm 2\%$  to  $94 \pm 2\%$  DPPH inhibition).

Levels of citric acid and pectin influenced colour, texture and antioxidant activity as well as retention and formation of aromatic compounds, especially in fructose–isomaltulose products. Correlations via a PLS were found between the aromatic profile of the products after storage and some of their quality parameters such as texture, colour and antioxidant content. Future research might involve correlation and identification of specific volatiles with different quality parameters.

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the formulation parameters (mostly pectin and citric acid levels), and their interactions, on the volatile fraction changes and aroma retention in the food gel was found. Two different phenomena could occur as a consequence of processing: (a) the modification (reduction or increase) of the compounds responsible of the aroma of fresh strawberry, and (b) the generation of new aromatic compounds. Some steps involved in processing of spreadable products, e.g. cutting or blending, sugar addition, etc., can lead to an increase in the activity of enzymes responsible for the development of new aromas. This fact has been also reported for many fruits under different condition such as osmotic stress, UV light, pH variations or under contact with metal ions (Escriche, Chiralt, Moreno & Serra, 2000; Olías, Pérez, Sanz & Ríos, 1993; Zabetakis & Holden, 1997).

In our previous study, 13 new compounds (6 aldehydes, 1 alcohol, 4 furans, 1 terpene and 1 nitrile) were found in strawberry spread-product after 24 h of processing, compared to raw material. It has been previously reported that the generation of new





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aldehydes might be related to the bio-oxidation of lipids as a consequence of wall disruption (Yilmaz, 2001). Eucalyptol, the only generated alcohol, appears during the heat treatment of fruit juices as a consequence of the hydrolysis of their corresponding glycoside precursors (Barron & Etiévant, 1990). The activation of Maillard reactions, or the catabolism of carotenoids and non-saturated fatty acids lead to furans generation (van Boekel, 2006). In the studied spreads, the type of sugar influenced the concentration of furfural (known as the most representative furan related to cooked flavours) (van Boekel, 2006; Varlet, Prost, & Serot, 2007). From the 29 original volatile compounds detected in fresh strawberry. 5 of them completely disappeared and 24 compounds (mostly esters and alcohols) varied their concentration after processing. The retention of typical fresh strawberry volatile compounds was very influenced by the blend of sugars, as well as percentage of pectin and citric acid; the higher the percentage of both, pectin and citric acid, the higher the retention of volatile profile of fresh strawberries, especially in those spreads formulated with isomaltulose-sucrose. The strengthen of gelling structure increases with increasing amounts of pectin, and citric acid, therefore, the gel has a closer structure where the exposure to external factors and the possible enzymatic reactions, which induce changes in aroma profile, are minimised. The role of the food matrix in controlling flavour retention, and even their release, has been previously pointed out (Boland, Delahunty & Vanruth, 2006; Druaux & Voilley, 1997).

It is well known that volatile profile can continue evolving with storage time. Nevertheless, a few number of studies have investigated the effect of storage conditions on aroma changes of fruity products such as juices, jams, purées, etc. (Kopjar et al., 2008; Torres, Chiralt, & Escriche, 2012). The majority of works have studied the effect of postharvest conditions such as temperature, relative humidity, controlled-atmosphere, etc. (Ayala-Zavala, Wang, Wang & González-Aguilar, 2004; Berna et al., 2007; Mo & Sung, 2007; Nielsen & Leufven, 2008; Pelayo, Ebeler & Kader, 2003; Vandendriessche et al., 2013), or the influence of osmotic treatment (Blanda et al., 2009; Rizzolo, Gerli, Prinzivalli, Buratti & Torreggiani, 2007) on the modification of strawberry aroma. Because foods are generally consumed after a certain time of storage, the evaluation of the changes taking place during this period would be also suitable. Since it is also expected that physicochemical properties such as mechanical and optical ones change, there is a need to better understand the link between flavour profile and texture or colour parameters evolution along storage, so colour and texture determinations might help to control aroma changes. In this sense, this study analysed the changes in the volatile profile occurring after 90 days of storage of 52 different strawberry spread-products formulated with healthier sugars to replace sucrose partially or totally. Additionally, the correlation between the aromatic compounds, some physicochemical properties (pH, water activity, texture and colour) and antioxidant activity as well as anthocyanin content were studied by means of a partial least squares regression (PLS regression).

#### 2. Material and methods

#### 2.1. Raw material

For this study, 15 different batches of raw strawberry (*Fragaria vesca*, Camarosa) were acquired from a local supermarket from February to June 2010. A selection based on colour, shape and ripeness were performed for each batch in order to reduce the variability of the raw material. Each different batch was enough to produce 3 or 4 different spreadable products, depending on the

#### Table 1

Independent variables and their level used for central composite design.

Independent variables	Symbol <sup>a</sup>	Coded variable levels				
		-2	-1	0	1	2
Isomaltulose (%) <sup>b</sup>	<i>X</i> <sub>1</sub>	0	12.5	25	37.5	50
Pectin (%) <sup>c</sup>	$X_2$	0.5	1	1.5	2	2.5
Citric acid (%) <sup>c</sup>	X3	0	0.25	0.5	0.75	1
Heat treatment time (min)	$X_4$	0	5	10	15	20

<sup>a</sup> Symbol with which each independent variable is cited in the text.

 ${}^{\rm b}$  % of isomaltulose in the total amount of sugar mix (sucrose-isomaltulose or fructose-isomaltulose).

<sup>c</sup> in final product.

batch. Then they were cut and washed in chlorinated water to eliminate possible field residues.

#### 2.2. Experimental methodology

Strawberry spread-products of 50 °Brix were formulated according to a statistical central composite design 2<sup>4</sup>+start (Peinado et al., 2013, 2015). For the experimental design, four independent variables were taken into account: percentages of isomaltulose, pectin and citric acid, as well as time of heat treatment (Table 1). Concretely, two groups of spread-product were obtained following two replicates of the above statistical design: one group containing 26 spread-products formulated with isomaltulose-sucrose, and another one of 26 spread-products formulated with isomaltulosefructose. The ingredients in the spreadable strawberry products were: strawberries, sugars (sucrose or fructose, and isomaltulose), pectin (as gelling agent), citric acid, and potassium sorbate (as microbiological preserver): the formulation of the products has already been explained in detail and was the implementation of dry osmotic dehydration studied previously (Rosa, Peinado, Heredia & Andrés, 2008; Peinado et al., 2013, 2015). Basically, ingredients were directly mixed in the correct proportions to reach the established concentration of 50 °Brix, avoiding therefore the typical equilibrium stage which takes place during osmotic dehydration. The quantities of raw strawberry and sugar were calculated according to the correspondent mass balance and they were dependent on the soluble solids content in the raw strawberries. Once the ingredients were mixed, the product was heated until it reached 85 °C. This temperature was necessary so the gel structure would not break up during storage as well as to make "hot canning" effective. Then, the product was placed in glass jars and some of the samples, depending on the statistical design, were heated for 5, 10, 15 or 20 min in a bath of boiling water. Finally, they were stored at 20 °C and darkness for 24 h and 90 days and after that, the correspondent analyses were performed.

#### 2.3. Analytical determinations

All analytical determinations were performed in triplicate after 24 h and 90 days of storage.

#### 2.3.1. Volatile profile characterisation

Following the same methodology as Peinado et al. (2013), volatile compounds were extracted by purge and trap thermal desorption using 2-pentanol as an internal standard. GC–MS analyses were performed using a Finnigan TRACETM MS (TermoQuest, Austin, USA) equipped with a BP-20 capillary column (SGE, Australia) (60 m × 0.32 mm i.d. × 1.0 mm film thickness). Helium at 1 ml min<sup>-1</sup> was used as a carrier gas. The ramp temperature was from 40 °C (2 min hold time) to 190 °C at 4 °C min<sup>-1</sup> and finally to 230 °C at 10 °C min<sup>-1</sup>.

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