Trehalose as a drying aid of fruit products: influence on physical properties, sensory characteristics and volatile retention

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ABSTRACT

The consumer acceptability of food products is influenced by many factors, including aroma, flavor, color and texture. In dehydrated fruit products the kind of dehydration process as well as the use of drying aids – such as sugars – can influence those attributes. Sucrose has been widely reported in literature as a drying aid of fruit pieces/ purees. However, the use of sucrose has important drawbacks such as its relatively low glass transition temperature (T_g), its susceptibility to hydrolysis in acidic media, participation in Maillard's reactions and a very sweet taste which somewhat masks the natural fruity flavor. Trehalose may be a potential substitute for sucrose in food formulations because; although its chemical structure is very similar to that of sucrose, it is more stable at low pH and high temperatures, it is not involved in caramelization and Maillard's reactions and its sweetness is only 45% of that of sucrose. Moreover, its T_g is much higher than that of sucrose thus improving physical stability of dried products.

This review discusses the potential use of trehalose as a functional replacement of sucrose as a drying aid to produce dried fruits; its utilization in carrier formulations to produce spray-dried encapsulated aromas is also discussed to some extent. Special attention is paid to the characteristics of aroma retention when trehalose is used as drying aid. Future trends are also discussed.

1. Sucrose: a traditional drying aid for fruit products

In dehydrated fruit products, the kind of dehydration process (i.e., air drying, freeze drying, etc.) as well as the use of drying aids - such as sugars - can have a great influence on sensory attributes like aroma and flavor among others. Drying methods involving high temperatures can lead to the loss of flavor compounds and promote reactions such as caramelization and the Maillard's one which may also alter the final product.

Freeze drying has proved to be the most suitable method for drying thermosensitive substances, minimizing thermal degradation reactions while relatively high aroma retention is attained in an amorphous carbohydrate microstructure matrix [1; 2; 3]. Historically, one approach to improve the quality of dried fruits (including freeze dried), consisted in the addition of sucrose prior to the dehydration process in order to increase aroma retention entrapping the organic volatiles in an amorphous dried matrix; and also to improve texture of the dried product. In the case of fruit pieces, sucrose has been incorporated through the use of the so-called "osmotic dehydration".

In the osmotic dehydration fruits are soaked in concentrated sucrose solutions. This process gives rise to at least two main simultaneous counter-current flows: a substantial water flow out of the food into the solution and a lower transfer of solute (sugar) from the solution into the fruit [4; 5; 6]. After additional drying (either air drying or freeze drying) the fruit pieces can be used as component of dry mixtures, i.e. cereals or snacks for direct consumption [7]. Collignan *et al.* [8] compared hot air drying of fresh and partially dehydrated apple, banana, mango and apricot, and found that the osmotic treatment before drying improved the sensory quality of the final product. Konopacka *et al.* [7] showed that sensory attributes were influenced significantly by the osmotic agent used for fruit impregnation before drying; dried fruits treated with sucrose were characterized by a predominantly sweet taste. The use of trehalose -instead of sucrose- may improve the quality of the dried fruit due to its reduced sweetness as compared to sucrose. Osmotic drying employing trehalose has been performed in apple slices [9] and strawberries [10].

Maltodextrins (MD) of relatively low dextrose equivalent (DE) have also been used as drying aids of fruits but mainly to increase the glass transition temperature of the dried product, thus improving its physical stability (i.e. stickiness) [11]. In this regard, sucrose has a main drawback, namely, its relatively low T_g which adversely affects the stability of the dried product; i.e. 60 ºC as compared to 110 ºC for trehalose [11].

2. Main sensory and physicochemical properties of trehalose as compared to sucrose

Trehalose is a non-reducing disaccharide composed of two glucose molecules linked by an α, α-1,1 glycosidic bond (Figure 1).

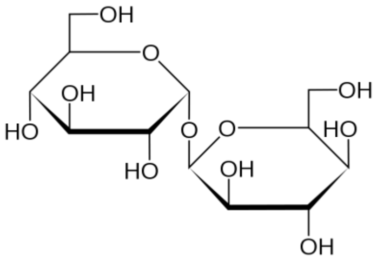

Figure 1. Trehalose molecule

This sugar is naturally found in mushrooms, yeasts, certain spores and even insect hemolymph. Its physical, chemical and biological properties have been studied over the past years. Trehalose has become increasingly available for food application being considered a “generally recognized as safe” (GRAS) multipurpose ingredient for all uses in food in accordance with current good manufacturing practices [12].

Even though its chemical structure is very similar to that of sucrose, trehalose has an extraordinary chemical stability, which makes it particularly interesting as a functional food additive. It is more stable than sucrose at low pH and high temperatures. It is poorly involved in caramelization process or the Maillard’s reaction with amino acids and proteins, thus preserving the natural color of the product. The water activity (a_w) of aqueous trehalose solutions is identical to that of sucrose at the same concentrations [13]. The latter property allows its use in combination with sucrose to optimize sweetness without altering the a_w, which is related to microbial stability.

The high glass transition temperature (T_g) of trehalose has been studied, in relation to its capacity to protect dried enzymes and microorganisms [14]. Table 1 shows the glass transition temperatures of some amorphous anhydrous carbohydrates.

Table 1 – T_g values for some amorphous carbohydrates (anhydrous)

	T_g (ºC)*
MD DE 10	160
Trehalose	115
Maltose	84
Sucrose	62
Glucose	37
Fructose	5

* Crowe *et al.*, [15]; Roos, [11].

Trehalose has a mild sweetness equivalent to 45% of sucrose [16]. Galmarini *et al.* [17] studied the sweetness of trehalose by describing its sweetness dynamic profile (time intensity (T-I) curves and gustatory reaction time (GRT)). These authors have reported the dynamic sweetness perception of commercial food grade trehalose, sucrose solutions and their mixtures at several concentrations. Trehalose had longer GRT with the range of concentration studied. Both sugars presented similar values of persistence and times of plateau and maximum intensity, whereas a significant difference was observed in intensity and GRT at equal concentrations. Trehalose had longer persistence than sucrose in equi-sweet solutions. The overall sweetness profile of sucrose solutions were perceived as similar to mixtures of sucrose/trehalose or solutions of trehalose alone (i.e., 29.9% sucrose solution and 0.6 sucrose:trehalose ratio mixture at 36.8% total solids), which suggests the possibility of sugar replacement without completely modifying sweetness perception.

According to literature data, trehalose, when tested as an ingredient in sports beverage supplement, elicits a lower insulin response than glucose [12].

3. Use of trehalose as a substitute of sucrose as a carrier for spray-dried encapsulated aromas

Spray drying is the most common technique to produce flavor powders from food flavor emulsions. Recipes for spray-dried flavors contain, in addition to the liquid flavor, carrier materials. Carbohydrates (maltodextrin, starch, arabic gum, sucrose) are the most common matrices used to entrap volatiles. It has been observed that depending on the aromatic compound and the carrier, entrapping efficiency can vary [18]. For flavor encapsulation, ingredients are mixed, emulsified/homogenized and spray dried; water content is reduced to below 5% and the flavor is encapsulated in an amorphous glassy carbohydrate matrix.

An ideal spray-drying carrier should have a high degree of solubility, limited viscosity at the 35-45% solution solids, emulsifying characteristics, good drying properties, non-hygroscopic character, bland taste, non-reactivity, and low cost. For these reasons, maltodextrins are commonly used for this type of spray drying processes. Besides, and due to the high glass transition temperature (T_g) of low DE maltodextrins the dried powder obtained is highly stable[19; 20]. Recent work done on strawberry and orange spray dried powder flavors showed that the presence of sucrose in the carrier formulation (MD and sucrose) reduced the physical stability of the final products [21]; and this was attributed to a reduction in the glass transition temperature when 40% of sucrose was incorporated in the dry carbohydrate matrix. As mentioned before, the type of carbohydrate carrier also governs flavor retention during the spray drying process [22; 23]. For this reason, disaccharides such as sucrose or lactose are sometimes included with maltodextrin in commercial formulations to improve flavor retention characteristics, even when some loss of physical stability of the product occurs.

Galmarini *et al.* [24] studied the sensory aroma profiles (sensory analysis and electronic nose) of orange oil encapsulated in different spray-dried carbohydrate matrices, composed of maltodextrin (MD) and different combinations with sucrose, trehalose, lactose, modified starch and Arabic gum. These authors demonstrated that the matrix composition determined the aromatic profile of spray-dried encapsulated orange flavors.

4. Instrumental methodology for the study of aromas: SPME-GC-MS

Over the last decades, more analytical and scientific methodologies have been developed for the study of flavors. Thus, the qualitative flavor composition of food products can now be well defined. In this sense, the application of these techniques has been a main goal to evaluate the influence of different food aids in the retention of volatiles.

Headspace sampling is a commonly technique used for the isolation of volatiles in solid and liquid samples. This methodology is based on the analyte partitioning between the sample matrix and the headspace. Volatile analytes are trapped from the headspace using dynamic or static procedures and analyzed by gas chromatography. The solid-phase microextraction (SPME) sampling technique has become a widely used tool for sample preparation for the analysis of volatile mixtures as an effective alternative to traditional methods for headspace sampling. SPME was originally developed for the analysis of pollutants in waters, however, and due to its effectiveness, its use was later extended to the analysis of volatiles in beverages and in high-water content solid samples like fruits and vegetables. This technique consists of two steps: (1) adsorption of the volatile analytes present in the headspace of the sample contained in standard headspace vials onto a coated-fused-adsorbent-silica fiber, which is part of the syringe needle and (2) thermal desorption of the analytes into the gas chromatograph injection port. The parameters affecting the SPME process were studied and compared by many authors [25; 26] and the influence of fiber coating in HS-SPME-GC [27 and cites therein].

Our group has evaluated the volatiles of freeze-dried strawberry purees using HS-SPME combined with GC coupled to a mass spectrometer (HS-SPME-GC-MS) [28]. For the sampling of volatile compounds from the headspace, a Supelco fiber holder with a 100 μm polydimethylsiloxane (PDMS) coated fused-silica fiber was used. Parameters affecting sample preparation and fiber exposure were reproduced according to Jetti *et al.* [26]. The GC-MS analysis of the volatiles was carried out on a Perkin Elmer Clarus 500 apparatus with a special configuration, consisting of one injector (split/splitless) connected by a flow splitter to two capillary columns: a) polyethylene glycol MW *ca*. 20.000 and b) 5 % phenyl-95%-methyl silicone, both 60 m x 0.25 mm with 25 μm of fixed phase (J&W Scientific). The polar column was connected to a FID, whereas the non-polar column was connected to a FID and a quadrupolar mass detector (70 eV) by a vent system. During sampling time (3 min), the injector was set in the splitless mode [29]. This system configuration enabled the obtention of three identification parameters of the volatiles introduced in the GC in a single run: retention indexes (RI) in both polar and non-polar columns as well as the mass spectra of each compound. The identification was performed by comparing mass spectra using the usual libraries [30; 31] and from the retention indices (relative to C_8-C_{24} *n*-alkanes) obtained in both columns and compared with those of reference compounds or published data [30; 26; 32].

For the comparison of the volatiles retained after freeze drying with the addition of different carbohydrates, the relative percentage contribution of the compounds was calculated from the total ion chromatograms by a computerized integration, assuming that all the responses factors were 1. In order to compare absolute values of volatiles retained after freeze drying in the different matrixes with the same percentage of strawberry puree, an internal standard was added in each run. Based on previous studies [33;26], a synthetic matrix was used, made of 4 g pectin, 23 g glucose, 23 g fructose, 10 g sucrose, 7 g citric acid and 1 g malic acid dissolved in 1 L of millipore water. A solution of 3-octanone and ethyl butyrate in methanol was used as internal and external standards respectively. External and internal standard solutions were added to 10 g of the synthetic matrix to obtain final concentrations of 6.25, 12.5 and 25 ppm of such volatiles. Volatile compounds were extracted using the same SPME fiber under the same conditions. Each analysis was carried out in duplicate. The GC running conditions were the same as those used for the strawberry puree samples. A calibration curve was used to calculate the concentration of the volatile compounds identified in the samples, assuming all of the responses factors were similar to that of ethyl butyrate.

5. Utilization of trehalose to improve sensory and physicochemical characteristics of freeze-dried fruits

Galmarini *et al.* [34] have found that the addition of trehalose improved physicochemical and sensory characteristics of freeze-dried strawberry purees in comparison to sucrose. They found that the addition of 40% trehalose led to a strawberry puree with a higher T_g (22.5 ºC onset value compared to 5.7 ºC obtained by the addition of equal amounts of sucrose). When stored at 32% relative humidity (RH) at room temperature (about 23 ºC) the sample containing trehalose experienced 5% radial shrinkage whereas the freeze-dried sample containing sucrose collapsed immediately. These authors also showed that the use of trehalose (instead of sucrose) as drying aid, helped to maintain a good sweetness/sourness balance, which was positively correlated to consumer preference. Kopjar *et al.* [35] investigated the influence of the addition of trehalose on textural properties of freeze dried strawberry pastes and found that the addition of trehalose influenced the hardness and stickiness of the samples.

Kopjar *et al.* [36] have studied the influence of the addition of trehalose on storage conditions and the quality (color, anthocyanin content, free radical scavenging activity, aroma compounds by HS-SPME-GC, and texture) of evaporated and freeze-dried strawberry cream fillings during storage at room temperature. They found that the addition of trehalose had a positive effect on color and anthocyanin retention, being the extent of retention proportional to the amount of trehalose added (3, 5 and 10%). The addition of trehalose also had a positive effect on fruity esters retention; however the amount of esters was not proportional to the amount of trehalose added thus concluding that the retention of aroma compounds depended not only on the process selected, but also on the structure of the aroma compounds. Komes *et al.* [37] freeze-dried pear purees added with sugars (sucrose and trehalose) and reported that the best retention of aroma compounds was achieved when trehalose was used. In dehydrated pear cubes, previously dipped in trehalose solution, the highest aroma retention was also determined.

Komes *et al.* [38] have compared the influence of the addition of different sugars on volatile retention during both freeze drying and foam-mat drying of strawberry puree. They added 8% of trehalose or sucrose before drying and observed that the addition of trehalose resulted in the lowest loss of total aroma as well as of individual fruit volatiles, as compared to the addition of sucrose, by using HS-SPME-GC-MS and HS-SPME-GC-FID.

The results obtained by Komes *et al.* [39] working with dehydrated apricot puree provided further supporting evidence for the ability of some sugars, especially trehalose, to increase the retention of volatiles responsible for the characteristic flavor of fruit products during dehydration. In this study, the purees dried without trehalose were more difficult to reconstitute and had lost much of their fresh flavor.

As mentioned before, the use of certain drying aids (carbohydrates) may improve the quality of dried fruits both by increasing aroma retention and also by rising the glass transition temperature (T_g) of the dried product. A high T_g value contributes to minimize physical changes such as shrinkage, collapse and/or crystallization of freeze-dried products [11]. It is well known that collapse may occur during storage of amorphous food structures; this physical change is time-dependent and is a function of ($T-T_g$), where T is the storage temperature [40]. Galmarini *et al.* [34] have studied collapse, which they arbitrarily defined as the relative percentage reduction in sample diameter after two weeks storage at 32% RH (at room temperature). As expected from measured values of T_g, the radial shrinkage was lower in samples containing trehalose and/or MD as compared to samples containing only sucrose. For example, plain strawberry puree experienced 20% radial shrinkage, whereas samples with 30% trehalose and 30% sucrose showed a radial shrinkage of 7.3 and 11.4%, respectively. Addition of MD, either alone or in combination with sugars, was more effective in preventing shrinkage, which could be predicted by its higher T_g.

6. Characteristics of aroma retention when trehalose is used as drying aid

Galmarini [28] has reported results obtained by HS-SPME-GC-MS for different samples (Table 2) of freeze-dried strawberry puree; they are presented as relative percentage area for each organic volatile in Table 3.

Table 2. Composition of strawberry purees systems.

Sample	Composition (g carbohydrate added/100 g total basis)
A	Plain fresh strawberry puree
B	Strawberry puree + 40% maltodextrin (MD)
C	Strawberry puree + 30% sucrose
D	Strawberry puree + 30% trehalose
E	Strawberry puree + 10% MD y 30% trehalose

In that study a total of 31 organic volatiles were detected, those which were identified had already been reported in literature as characteristic of strawberry [40; 31; 41; 42; 37; 26]. Esters were the most abundant compounds by far; terpenes (linalool and nerolidol), a lactone (γ-dodecalactone) and five unidentified compounds (U1 to U5) were also found. The relative percentage area (obtained for each compound in the freeze-dried samples) was different ($p<0.05$) for most volatiles except for *(E)*-2-hexenyl acetate, hexyl hexanoate, octyl-2-methyl butyrate and U1 (Table 3). Plain fresh strawberry puree and reconstituted freeze-dried puree added with trehalose, or trehalose and MD, had volatiles of high and low molecular weight. On the other hand, freeze dried samples added with MD or sucrose (B and C) mainly retained high molecular weight compounds.

Figure 2a, shows the headspace concentration (ppm) of the different compounds in the reconstituted freeze dried strawberry added with 30% trehalose (D) or with 30% sucrose (C). Qualitative and quantitative differences were found between these two samples. The trehalose treated sample had significantly higher concentration of: methyl butyrate, ethyl butyrate, methyl hexanoate, hexyl acetate, ethyl octanoate, octyl acetate ($p<0.001$), ethyl decanoate ($p<0.01$) and decyl acetate ($p<0.05$). The greater differences were observed for ethyl hexanoate (concentration almost five times bigger in sample D than in C) and in octyl acetate (concentration being six times higher in sample D than in C). Even though the concentration of ethyl octanoate was not relevant compared to the rest of the volatiles, its concentration was eight times higher in the trehalose freeze-dried sample. Galmarini [28] have also reported that freeze dried puree added with sucrose had a higher concentration of hexyl hexanoate, octyl butyrate, γ-dodecalactone ($p<0.05$) and (E)-nerolidol ($p<0.01$). The sucrose treated sample retained 2.4 and 2.2 times more octyl butyrate and (E)-nerolidol than trehalose one, and had 9 times more γ-dodecalactone (although the latter volatile only represented a small fraction of the total profile).

Figure 2.b shows a comparison between the quantitative volatile composition (in ppm) of freeze dried purees added with 40% MD (B) and 30% trehalose plus 10% MD (E). Although every compound had a higher concentration in sample E, differences were significant only for: ethyl hexanoate, hexyl acetate, octyl acetate (all $p<0.001$), ethyl butyrate, linalool, ethyl octanoate, decyl acetate (all $p<0.01$), hexyl hexanoate, ethyl decanoate, octyl pivalate, *(E)*-nerolidol, octyl hexanoate and ethyl dodecanoate (all $p<0.05$). As found for sample D, the higher volatile concentrations corresponded to ethyl hexanoate, octyl acetate, octyl hexanoate and octyl butyrate. The combination of trehalose plus MD caused a greater volatile retention than the equal amount of MD. Even when the ratio of puree in sample E was smaller than in samples C and D (60% *vs.* 70%), sample E had a higher concentration of total volatiles.

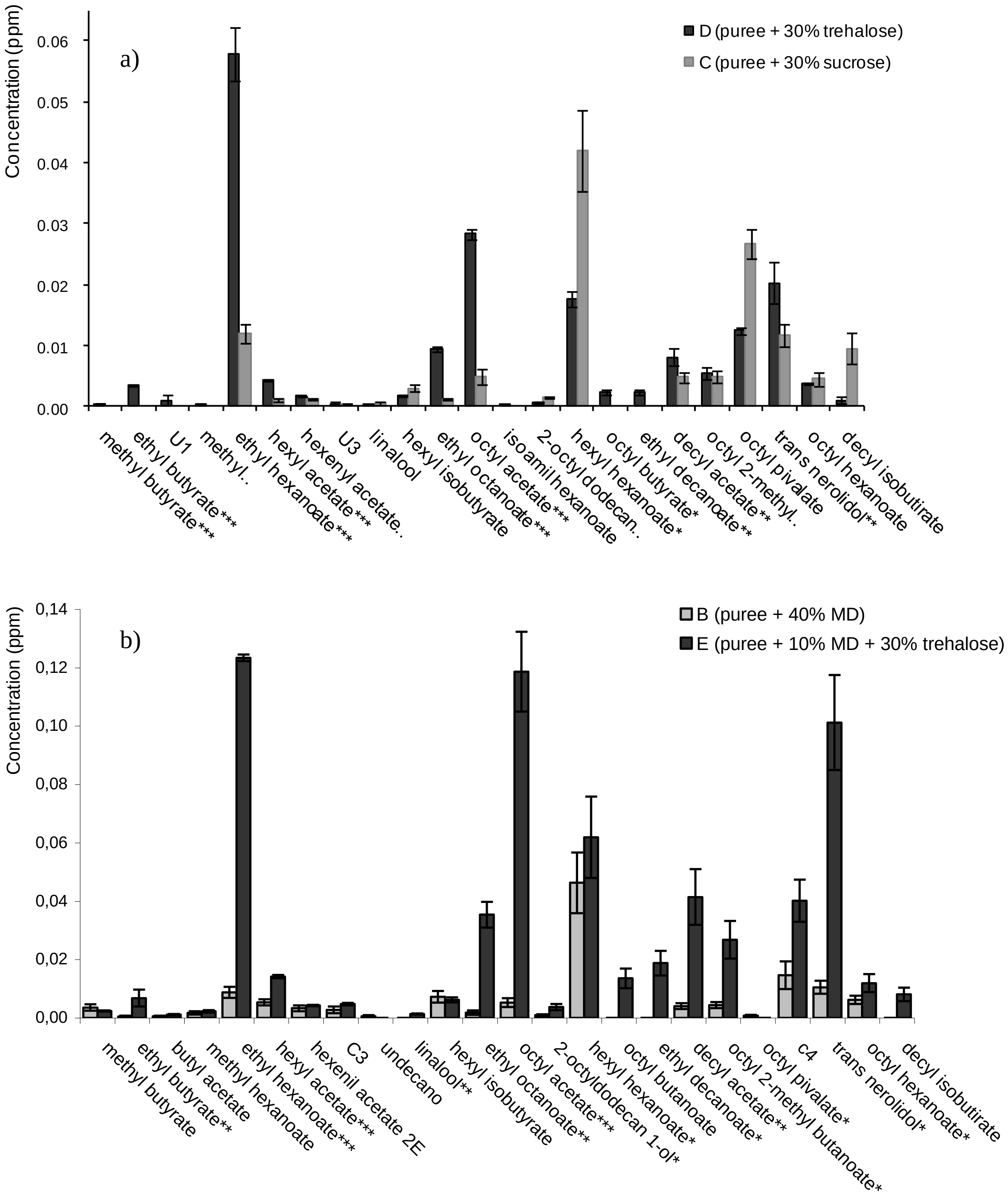

Figure 2. Comparison of volatile concentrations in freeze dried strawberry purees, a) 30 % sucrose *vs* 30 % trehalose; b) 40 % maltodextrin (MD) *vs* 10 % MD + 30 % trehalose. $p<0,001$; ** $p<0,01$; *$p<0,05$; (Student–Neuman–Keuls (SNK) test).

Galmarini [28] has also compared sensory and chromatography data; the relationship between volatiles determined by HS-SPME-GC-MS and the sensory attributes are shown in Figure 3. The first two factors explained 62.7% of the variance between sensory attributes (X) and chemical identified compounds (Y).

The sensory attributes, syrupy, caramel and banana flavor all correlated with the organic volatiles methyl butyrate, isoamyl hexanoate and ethyl 3-methyl butyrate (quadrant I, Figure 3). Methyl butyrate and isoamyl hexanoate are described in literature (Table 3) as banana, sweet and fruity smells. In a descriptive study of strawberry aroma [40] the smell of ethyl 3-methyl butyrate was described as cooked apple. This would explain the relationship found between ethyl 3-methyl butyrate and the "cooked" attribute (quadrant II, Figure 3). The sensory attributes "peach", "milky" and "strawberry candy" were related to *(E)*-2-hexenyl acetate, butyl acetate, ethyl butyrate and *(E)*- nerolidol. 2-*(E)*-Hexenyl acetate has been related to peach aroma, among other fruity ones (Table 3) whereas ethyl butyrate was related to "fruity", "sweet", "strawberry", "butter" and "vanilla" aromas.

The attributes "green strawberry", "pungent", "grassy" and "leathery" located in quadrant IV (Figure 3) were opposite to the previously mentioned attributes characteristic of ripe fruit, such as caramel, banana, syrupy, peach and milky. The organic volatiles associated with quadrant IV were octyl pivalate, octyl hexanoate, ethyl decanoate and octyl 2-methyl butyrate, all of them corresponding to the waxy aromas family.

The attributes "pineapples", "fruity", "apple", "citric" and "floral" are grouped in quadrant II (Figure 4) and are highly desirable descriptors in a strawberry puree. The volatiles related to these attributes were linalool, γ-decalactone and the fruity esters hexyl acetate, ethyl hexanoate, ethyl dodecanoate, ethyl octanoate and octyl acetate. Ethyl hexanoate and γ-dodecalactone have been reported as volatiles with a fruity, sweet, strawberry aroma; being ethyl hexanoate also related to apple and pineapple (Table 3). Octyl acetate, has been related to floral, jasmine, orange and herbaceous aromas (Table 3) which explains its high correlation with the attributes floral, citric and, in a smaller degree, grassy (quadrant IV, Figure 3). The ethyl octanoate has been associated to floral and apple aromas and ethyl dodecanoate to floral one.

"Cooked", "prune", "burnt", "stale", "sulfur" and "musty" are non-desirable attributes and were located opposite to the descriptors and volatiles mentioned above (quadrant III, Figure 3). The organic volatiles which correlated with these descriptors were methyl hexanoate, hexyl isobutyrate, octyl butyrate, undecane, hexyl hexanoate, decyl isobutyrate and ethyl 3-methyl butyrate.

Galmarini [28] has concluded that the type of carbohydrate (trehalose, sucrose or maltodextrin) used as a drying aid significantly modifies the type and concentration of the volatiles retained during freeze drying, as well as the aroma profile. The sample added only with trehalose showed the chromatographic and sensory profile most similar to the fresh strawberry puree. The use of sucrose (instead of trehalose) led to a similar sensory profile but induced a different volatile retention, as determined by HS-SPME-GC-MS.

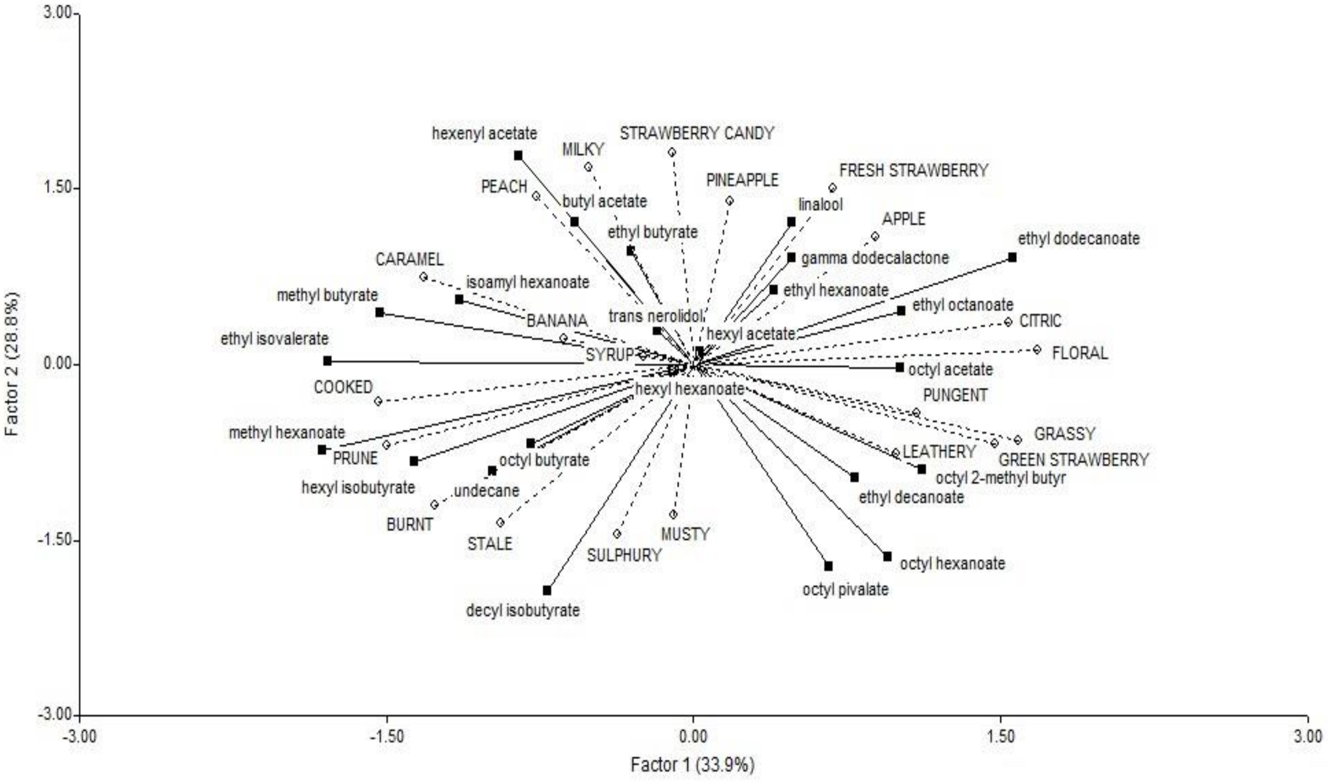

Figure 3. Relationship between volatiles determined by HS-SPME-GC-MS and sensory attributes

Prospects

This review showed possible applications of trehalose as a potentially beneficial food ingredient, with the aim to improve the quality of dehydrated fruit products as compared to conventional methods which use sucrose as additive. The beneficial effects of trehalose are mainly manifested through a rise in the glass transition temperature (T_g,), a better aroma retention, and an adequate sweetness/sourness balance, while allowing a better perception of the natural fruit flavor. Further work will be needed to fully understand the mechanisms underlying the effects observed.

Table 3. Peak area ratios of aroma compounds in the freeze-dried and fresh strawberry purees. Retention Index (RI) values for identified compounds are listed along with the aromatic descriptor found in literature.

Nº	Compound	RI*	RI**	A***	B	C	D	E	Odour descriptor
1	Methyl butyrate	728	986	4.011 a	2.616 b	0.674 c	0.297 c	0.721 c	Fruit, sweetish [1] Sweet, banana, apple [6]
2	Ethyl butyrate	804	1046	8.018 a	0.953 b	0.609 b	1.636 b	3.492 b	Fruit, sweetish, apple [1] Vanilla [4] Fruity, slightly buttery, sweet, strawberry[7]
3	Butyl acetate	811	1085	1.835 a	0.000 b	0.000 b	0.000 b	0.280 b	Fruity, banana[8]
4	Ethyl 3-methylbutyrate	854	1069	0.716 a	0.660 a	0.000 b	0.000 b	0.000 b	Fruit, sweetish [2] Cooked apple [4] Sweet, fruity, butter, banana [7]
5	Unknown1	875	-	0.074 a	0.000 a	0.000 a	0.144 a	0.000 a	
6	Methyl hexanoate	927	1197	0.738 ab	1.086 a	0.332 b	0.299 b	0.358 b	Fruity, sweetish [1] Fruit, fresh, sweet [3]
7	Butyl butyrate	994	1229	0.992 a	1.583 b	0.391 a	0.575 a	0.342 a	Fruit, sweet, pineapple [6]
8	Ethyl hexanoate	998	1244	34.539 a	1.310 c	0.207 c	25.030 b	25.782 b	Apple peel, fruity [3] Fruity/acid [4] Pineapple, strawberry [7]
9	Hexyl acetate	1009	1282	2.378 c	1.511 b	0.957 a	2.243 c	2.118 c	Fruit, sweetish [1] Fruit, herb [3]
10	*(E)*-2-Hexenyl acetate	1013	1340	1.246 a	0.967 a	1.168 a	0.944 a	0.761 a	Banana, fruity, peach, par, pineapple, strawberry, waxy [8]
11	Unknown 2	1054	-	0.077 bc	0.000 a	0.000 a	0.085 c	0.051 b	
12	Unknown 3	1062	-	0.220 b	0.000 a	0.895 c	0.039 a	0.000 a	
13	Linalool	1097	1553	0.798 a	0.300 a	2.278 b	0.127 a	0.449 a	Floral odour with citrus-like [2] Flower, lavender [3]
14	Undecane	1101	1100	0.044 a	0.260 c	0.169 b	0.127 ab	0.052 a	Alkane [3]
15	Hexyl isobutyrate	1151	-	1.205 ab	3.421 d	2.079 c	1.569 b	0.836 a	Fruity, ripe, sweet [4]
16	Ethyl octanoate	1197	1444	4.974 a	0.000 c	1.167 b	5.871 a	5.716 a	Fruity, apple, pineapple, floral [3]

17	Octyl acetate	1213	1483	13.698 b	2.787 a	2.610 a	16.641 c	19.477 d	Fruity, floral, herbaceous, orange, green, jasmine [8]
18	Isoamyl hexanoate	1246	-	0.286 c	0.182 b	0.000 a	0.305 c	0.000 a	Banana, fruity, green, waxy [6]
19	Hexyl hexanoate	1379	1617	0.353 a	0.797a	0.782 a	1.311 a	0.327 a	Green, green tea [2] Apple Peel [3] Fatty, with burnt notes [5]
20	Octyl butyrate	1387	1623	6.418 c	30.601 a	26.159 b	7.702 c	8.429 c	Musty, oily, earthy [8]
21	Ethyl decanoate	1398		0.815 bc	0.569 b	0.000 a	0.953 c	1.762 d	Sweet, oily, nut-like, yeast [5]
22	Decyl acetate	1408	1687	1.082 a	0.466 c	0.000 d	1.403 b	0.000 d	Sweet, fruity-oily, rose, wax [8]
23	Octyl 2-methyl butyrate	1435	1634	2.590 a	2.748 a	2.596 a	3.480 a	3.374 a	Waxy, fruity, green, musty [8]
24	Octyl pivalate	1440	-	1.865 a	2.913 b	2.706 b	2.369 ab	3.090 b	
25	Unknown 4	1470	-	0.055 a	1.382 b	0.000 a	0.176 a	0.044 a	
26	(*E)*-nerolidol	1563	2050	4.137 a	12.253 b	19.731 c	6.463 a	3.391 a	Sweaty [2] Wood, flower, wax [3] Green, fruity [4]
27	Octyl hexanoate	1570	1829	4.737 a	7.735 b	4.815 a	8.319 b	12.328 c	Sweet, green [2]
28	Decyl isobutyrate	1590	-	0.698 a	4.748 d	1.737 b	1.703 b	2.662 c	
29	Ethyl dodecanoate	1594	1827	0.375 a	0.000 a	1.395 b	0.581 a	0.838 ab	Mango like [3]
30	γ-Dodecalactone	1685	2396	1.101 a	2.233 a	14.223 b	2.569 a	0.609 a	Sweet, coconut like [2] Sweet, fruit, flower [3] Strawberry [4,9]
31	Unknown 5	2230	-	0.173 a	1.718 b	1.582 b	0.735 a	0.139 a	

Lower case letters represent significant differences ($p<0.05$). Student's test and a posteriori Student-Neuman-Keuls analysis.

RI*: Retention Index in non polar column.

RI**: Retention Index in polar column.

***Sample composition is presented in Table 1.

[1] Ulrich *et al.* [43]

[2] Fukuhara *et al.* [44]

[3] Acree and Arn [45]

[4] Gomes Da Silva and Chaves Das Neves [41]

[5] Fontarome chemicals [46]

[6] Frutarom Chemicals [47]

[7] Symrise Chemicals [48]

[8] The Good Scents Company [49]

[9] Shieberle and Hofmann [33]

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