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Aromatic profiles of spray dried encapsulated orange flavours: influence of matrix composition on aroma retention evaluated by sensory analysis and electronic nose techniques.

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Running title: Aromatic profiles of encapsulated flavours

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ABSTRACT

Spray dried orange flavour encapsulated in different amorphous matrices comprised of maltodextrin (MD) and different combinations with; sucrose, trehalose, lactose, modified starch and gum arabic (ga); were evaluated by sensory analysis and electronic nose (e-nose). With both techniques the flavours encapsulated in MD-sucrose and MD -lactose-sucrose were perceived as similar. However, the e-nose did not detect any differences among the other matrices (MD-trehalose, MD, MD-sucrose at a different concentration and MD-ga). On the contrary, sensory analysis was able to group MD-trehalose and MD describing them by: woody, marmalade, syrup, citrus terpens, and Vitamin C; MD -sucrose at 40% and 10% concentration and MD -lactose-sucrose were grouped and represented by powder juice, tangerine and pungency, while MD-ga was differentiated from the rest by the attributes peely, plastic, solvent and green. In this way, it was shown that matrix composition determines the aromatic profile of spray dried encapsulated orange flavours.

Keywords: Spray dried – Orange encapsulated flavours – Sensory profile – electronic nose – trehalose - sucrose

INTRODUCTION

Spray drying is the most common technique to produce flavour powders from food flavour emulsion; recipes for spray-dried flavours contain, in addition to the liquid flavour, carrier materials such as maltodextrin (MD), gum arabic and modified starch. Ingredients are mixed, emulsified/homogenized and spray dried; water content is reduced to below 5% and the flavour is encapsulated in an amorphous glassy carbohydrate matrix.

The requirements for an ideal spray-drying carrier include a high degree of solubility, limited viscosity at the 35-45% solution solids range, 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 applications. Also, the high glass transition temperature (T_g) of low DE maltodextrins provides good product stability to the dried powder (Beristain *et. al* 2002; Bhandari & Hartel, 2005).

It is well known that, among other factors, the type of carrier governs flavour retention during the spray drying process (Menting *et al*. 1970; Thijssen, 1971); and for this reason disaccharides, such as sucrose or lactose are sometimes included with maltodextrin in commercial formulations to improve retention characteristics.

However, the low T_g of sucrose (Roos, 1995) may adversely affect stability. Food powders containing amorphous carbohydrates can undergo several physical changes which lead to deterioration of quality (Roos & Karel, 1991). Recent work done on strawberry and orange spray dried powder flavours showed that the presence of sucrose in the carrier formulation (MD and sucrose) affected storage stability in a negative way (Busso Casatti *et al*., 2006)**,** because it reduces the glass transition temperature dramatically when 40 % of sucrose is incorporated in the dry carbohydrate matrix.

Trehalose is a naturally occurring non reducing disaccharide which consists of two glucose molecules linked in a 1,1-position by a α -glycosidic bond. In the last years the use of trehalose as a functional food additive has been approved in many countries. The glass transition temperature for trehalose is much higher than that of sucrose (115 ºC as opposed to 77 ºC for sucrose; Komes *et al*., 2003) which would contribute to the physical stability of spray dried matrices containing trehalose instead of sucrose. It is well known that during spray drying encapsulation of flavours partial loss of volatile compounds usually occurs leading to an alteration of the total volatile composition and/or a variation of the ratio of the different compounds in the spray dried product. Komes *et al*. (2003; 2005) observed that the addition of trehalose to dehydrated strawberry and apricot purees resulted in the lowest loss of total aroma as well as of individual fruit volatiles when compared to sucrose.

In present work aromatic profiles of orange oil encapsulated in different spray dried carbohydrate matrices are studied. Orange oil aroma is a complex mixture of various chemical components, primarily (+)-limonene (>90%) responsible for the base sensory character of the citrus oils. The aroma is determined by the aldehydes, mainly octanal and decanal, and both citrals and esters. The sesquiterpenes aldehydes, α and β sinensal contribute particularly to the specific sweet orange aroma (Wright, 1995). Even though odour mixtures bear a strong resemblance in their character to the quality characteristics of the individual components, no single compound possess the odour quality characteristic of the blend (Lawless & Heymann, 1998). In addition, the retention of aroma compounds during spray drying encapsulation is expected to depend on the interactions of aroma compounds and encapsulating components of the matrix. This results in changing the volatile profile and the sensory perception of aroma up on reconstitution of the flavour powder.

Unlike the traditional instruments (e.g. gas chromatography) which discriminate the different flavour components, electronic noses (e-noses) measure the aroma

components as a whole, thus resembling the human nose. However, it must be noted that the e-noses measure not total odour intensity, but rather give an undefined measure of the total volatiles, being them aromatic or not (Stephan *et al*., 2000). Some semi-specific metal oxides sensors do exist, but the ability to, for example, detect the character impact component of some aroma is still far away. In this way, it is important to compare the study with sensory analysis which gives a specific perception of a particular flavour while allowing a descriptive and a quantitative evaluation of the different aroma compounds. This comparative and complementary analysis was used by Baby *et al*. (1999) to determine odour intensities of different dilutions of powder orange juice using a sensory panel and an e-nose as measuring instruments, observing that both intensities could fit to similar mathematical functions.

The aim of this work was to compare the aromatic profile of spray dried orange flavours encapsulated in different carbohydrate matrices by means of sensory analysis and an e-nose. In addition, the efficiency of aroma retention of sucrose and trehalose (in a mixture with maltodextrin) were compared in order to explore the potential of this disaccharide as a carrier for producing spray dried flavours.

MATERIALS AND METHODS

Samples

Five commercial spray dried encapsulated orange flavours (samples 1 to 5) (5 % water content) and one sample specially prepared using trehalose were manufactured in a flavour company located in Buenos Aires, Argentina. Table 1 shows the composition of the spray dried flavours. Maltodextrin DE 12 (MD 12) accounts for most of the amorphous dried matrix in samples 1 and 2; while in samples 3, 4, 5 and 6 it has been partially replaced by one or the other of the following disaccharides: sucrose, lactose and trehalose. Samples 1, 3, 4, 5 and 6 also have a small proportion of modified starch in their composition while sample 2 has only MD and gum arabic.

The sample containing trehalose (number 6) was specially prepared for this study since this disaccharide is not locally commercially used for this purpose yet. Processing conditions during flavour oil emulsification (which can affect emulsion size) were similar for all samples; the same for operating conditions of the atomizer during spray drying, to minimize effects on particle size distribution of the spray-dried powder.

Sensory Analysis

Panel training

Twelve paid assessors (12 females, 19-23 years old) students of Facultad de Ciencias Agrarias, Pontificia Universidad Católica Argentina were trained in descriptive analysis of orange flavours (eight hours). During training period, judges performed the following tasks: 1) Odour identification using standard solutions (Table 2); 2) Attribute generation of the different orange samples with the aid of standards (Table 2), 3) Matching of aromas, 4) Use of unstructured scales.

Aroma profiling

The experiment was divided in two phases: 1º) Triangle Test (ASTM, 1977) was performed to compare orange flavours and develop information about the characteristics of the samples. During two testing sessions (2 hours long each), assessors were required to pick the sample which they believed was different and describe the attributes responsible for that difference. The powder flavour solutions (5% in distilled water) were placed (15 mL) in sealed glass flasks (35 ml capacity, 3 cm diameter opening) and presented to assessors identified with random three digit codes. Testing took place in individual booths kept at a temperature of 22 ± 2ºC and illuminated with red light in order to mask differences due to colour intensity. Descriptors which accounted for the differences were listed and their frequency of mention was analyzed in order to obtain a control sample for the descriptive analysis. This control sample was the one with the highest frequency of mention for the given attribute (Table 2).

2º) Descriptive Analysis was done by a combination of the quantitative descriptive analysis (QDA) method (Stone & Sidel, 1993) and the difference-from-control test (Meilgaard *et. al,* 1991). The aid of standards was provided in order to quantify the 14 attributes: freshly squeezed, juice powder, citrus terpens, vitamin C, tangerine, candy, solvent, plastic, peely, marmalade, woody, pungency, green, syrup. Six samples and a control - which was specific for each attribute - were presented in each session (seven sessions; two hour long each, aprox.); assessors did not know that the control sample was the same as one of the coded samples. For every descriptor judges were asked to sniff the headspace generated in the control sample and then quantify the given descriptor for the six samples by comparing aroma intensity with the control; samples were evaluated in duplicate. Evaluation was done only by sniffing the headspace in order to be able to compare results with those obtained with the e-nose.

E-nose Analysis

The E-nose used MOSES II (MOdular SEnsor System), Tübingen, Germany, was equipped with eight tin dioxide (SnO_2) sensors and eight quartz microbalance (QMB) sensors. Different doping of tin oxide sensors as well as different polymeric coatings of QMB sensors confer them higher sensitivity and special selectivity for certain gases. Surface resistivity of SnO_2 sensors changes according to the oxidant or reducing properties of the adsorbed gases. The quartz crystals of QMB are operated in an oscillator circuit and in the absence of analyte gas they oscillate with their usual frequency (12-14 MHz). If the polymer coating adsorbs selectively an analyte gas (adsorption is temporary and reversible) the crystal mass increases, thus reducing the frequency of vibration. In consequence, the frequency change results proportional to the analyte concentration (Mitrovics *et. al*, 1997).

In this experience only the oxide sensors were used since they allowed a better discrimination among samples. Solutions at 5% in water of the six samples were analyzed. For each sample three vials containing 3 ml of the solution were prepared,

incubated at 40 ºC for 15 minutes with a 20 minute interval between samples and placed in a headspace sampler Dani HSS 86.50. Volatiles were carried to the sensors by a stream of synthetic air (flow rate 30 ml/min).

Each analyzed specimen produced, consequently, a plot of sixteen curves (being each one the output signal of each sensor versus time). It is necessary to reduce the large quantity of data so as to describe each sample with a minimal number of parameters. The Principal Component Analysis (PCA) algorithm makes use of the advantage that sensors are relatively non specific and that it can combine the signals of all the sensors in a unique signal. Employing this method, similar odours tend to be grouped in clusters and the result is a bidimensional plot (axes are the components which contribute most to the odours expressed in %).

Statistical Analysis

The binomial distribution was used to calculate the significant level for the triangle test, based on a number of correct answers. Analysis of variance (ANOVA) was carried out to assess attributes significantly different among samples using the General Linear Model command in SPSS v. 13.0. The variability of each descriptor was studied using a model where assessor was considered a random factor and sample and replication fixed factors. Multiple means comparisons were carried out by Student-Neuman- Keuls (SNK) test at $p< 0.05$. PCA was conducted to examine the relationship among attributes and samples, correlation matrix was used and the minimum eigenvalue was set at 1. Clusters were performed by K-Means command in SPSS v. 13.0. E-nose used PCA as the statistical method to process the data obtained by MOSES II.

RESULTS

Triangle Test

Table 3 shows the results for the triangle test for all samples. As it can be seen all samples resulted significantly different except for sample 4 respect to sample 5. These two samples had sucrose in low concentrations (5 and 10% respectively) and the same amount of aroma (18%).

Analysis of Variance (ANOVA)

ANOVA of mixed model for attribute scores are summarized in Table 4. Sources of variation were samples, assessors and sample*assessor interaction. Replications and assessor*replication and sample*replication interactions were non significant supporting the interpretations that the use of attributes was consistent.

Effect of assessors was significant for only one attribute (vitamin C, $p<0.05$) and sample*assessor interaction was significant for two attributes (freshly squeezed, $p<0.01$ and vitamin C, $p<0.05$) indicating that not all the judges evaluated all the samples in the same fashion for those descriptors. Other authors observed that this can happen when samples are very similar in their sensory properties so assessors can not differentiate easily among them (Tang *et al.,* 1999; Zamora & Guirao, 2002). In order to verify this observation we examined the samples which were different from each other for these attributes. ANOVAs were calculated for the samples whose means had the biggest difference and which were found significantly different by SNK test (Table 5). No significant interactions were found for freshly squeezed (samples selected 1 and 4) $F_{1,11}=1.001$ and vitamin C (samples selected 1 and 5) $F_{1,11}=1.969$. For this last attribute no significant difference was found among assessors for the most different samples (1 and 5) $F_{1,11}= 0.886$. This data shows that for these two attributes there was an agreement among assessors. This being so, interactions (Table 4) could be attributed to the similarity among samples rather than to different criteria used by judges.

The means of attributes which showed significant differences among samples are presented in Table 5. Sample 1 presented the higher mean scores for the attributes: citrus terpens (75.3), vitamin C (65.9) and marmalade (46.4); sample 2 had

the higher mean for plastic (66.9); and sample 6 had the maximum mean value for woody (61.4) and syrup (72.2). As regards samples 3, 4 and 5, even though neither of them presented a maximum nor a minimum value for any attribute, all of them shared the maximum value for freshly squeezed and candy while 4 and 5 also shared the maximum for powder juice and pungency. These results are in accordance with triangle tests (Table 3).

Aroma Profiling

PCA explains 93.5% of the variance among samples with the first three components; the biplot of Principal Component 1 (PC1) vs. Principal Component 2 (PC2) is shown in Fig. 1. The most important attributes which compose PC1 are juice powder, freshly squeezed, pungency and candy opposite to woody, syrup and vitamin C; and PC2 is defined by plastic, solvent and green.

Three different clusters were formed; samples 3, 4 and 5 (MD-sucrose (40%), MD-sucrose (5%)-lactose (21%), MD-sucrose (10%) were grouped and described by the attributes juice powder, freshly squeezed, candy, tangerine and pungency. Samples 6 and 1 (MD-trehalose and MD) had mainly the aromas woody, marmalade, syrup, citrus terpens, and vitamin C. Finally, sample 2 (MD-gum arabic) was characterized by solvent, plastic, peely and green. Samples 4 and 5 are grouped one more time as shown in Table 5 and in the triangle test (Table 3).

E-nose

PCA performed with data obtained by the E-nose explains 99.2% of the variance among samples with the first component (Fig.2). Only two clusters can be observed: samples 4 and 5 are grouped together while the rest of the samples (1, 2, 3 and 6) cannot be discriminated, forming a whole different group.

DISCUSSION

Samples 4 and 5 are perceived as similar using either sensory analysis or e-nose evaluation. Both powder samples contained sucrose in low concentrations (5 and 10%, respectively) and the same amount of encapsulated aroma (18%) in their composition; in addition sample 4 contained lactose in its matrix (21%) but, apparently, lactose would not be contributing to the differential volatile retention during spray drying process. The e-nose could not detect any differences among samples 1, 2, 3 and 6. On the other hand, according to the sensory analysis, samples 4 and 5 were grouped together with sample 3 (Fig.1). These three samples were described by the same attributes but differed in the mean intensity of the descriptors mentioned, being sample 3 the one with the lowest intensity for powder juice, pungency, tangerine, solvent and plastic. Sample 3 also had sucrose in its composition, but in a higher concentration (40%) and had a smaller aroma fraction (13%).

The human nose was able to discriminate samples 1 and 6 from the rest (Fig. 1), describing them by the same attributes; however the e-nose did not find significant differences. These two samples (1 and 6) differed in matrix composition as well as in aroma proportion (sample 1: 72% maltodextrin, no disaccharide, 16% aroma; sample 6: 40% maltodextrin, 40% trehalose, 13% aroma). Finally, sample 2 was found different from all the rest (Fig. 1); this sample contained the most aroma (20%) and gum arabic instead of a disaccharide.

A difference between the human and e-nose is the pungency sensation measured only by sensory analysis. This sensation is mediated not by smell fibres, but by other chemo sensitive fibres as trigeminal nerve branches. Some compounds which are perceived as being purely olfactory (e.g. butyl acetate, a fruity odour) can elicit activity in the trigeminal nerve without creating sensations of burning or stinging (Cain,

1974). Electrophysiological and psychophysical evidence indicate that odours at concentrations lower than those generally considered to be non-irritating can stimulate both olfactory and trigeminal chemo receptors, and this stimulation can contribute to the perceived odour intensity (Maruniak, 1988; Delwiche, 2004). Since pungency is only detected by human nose this perception could contribute to differentiate both measurements (human and e-nose).

As regards substitution of sucrose by trehalose in the matrix composition, it was noted that samples 3 and 6 (MD-sucrose, MD-trehalose) had different aroma profiles suggesting that different volatiles are retained during spray drying according to the sugar used in the encapsulating matrix (sucrose or trehalose). Komes *et al*. (2005) studied the influence of the addition of trehalose or sucrose on the retention of volatiles in dehydrated apricot puree (foam-mat drying and freeze drying) and found that the best retention of aroma compounds was obtained when trehalose was added. It is to be noted, however, that in present work we used spray drying and also the volatile composition of orange flavour is likely to be quite different from that of apricot flavour.

CONCLUSIONS

Sensory analysis proved to be a more sensitive tool than the e-nose for grouping and describing the orange encapsulated flavours. Although both methods produced “coherent” results, by sensory analysis samples were discriminated more sharply. In addition, sensory analysis has the advantage that it can “name” the difference, this can not be done with the e-nose.

As expected, the composition of the matrix likely influenced the type and the amount of volatiles retained, as suggested by the PCA analysis. In this way, by changing the matrix composition used for aroma encapsulation different aromatic profiles can be obtained, which will give as a result different products. For example, if attributes such as freshly squeezed, pungency, tangerine, candy and juice powder are

desired in the final aromatic profile of an orange flavoured product, then the matrix used for encapsulation should include sucrose. On the other hand, if woody or marmalade notes are desired in the final product, trehalose should be used in the encapsulating matrix. Citrus notes are obtained by using a MD and modified starch matrix while a matrix with MD and gum arabic would be recommended for a product in which peely and green attributes are desired.

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Table 1: Composition (% dry basis) of spray dried flavours

Sample	Maltodextrin	Modified starch	Sucrose	Gum arabic	Lactose	Trehalose	Aroma
1	78	5	-	-	-	-	16
2	72	-	-	8	-	-	20
3	40	4	40	-	-	-	13
4	50	6	5	-	21	-	18
5	64	7	10	-	-	-	18
6	40	4	-	-	-	40	13

Table 2. Definition, attribute and standard recipe used by the trained panel to describe orange flavours.

Attribute	Standard recipe	Definition	Control Sample
Freshly squeezed	Filter paper soaked in pure orange essence oil (“Fresh orange”, *Firmenich*, Argentina), placed in a sealed glass flask.	Aroma evocative of natural, fresh, recently prepared orange juice.	3
Juice powder	50 g of juice powder (“orange”, *Tang*, Argentina) reconstituted in 100 ml of distilled water.	Related to the aroma of orange juice obtained from reconstituted juice powder.	5
Citrus terpens	Filter paper soaked in Gamma terpinene essence oil; (“Gamma terpinene”, *IFF*, Argentina) placed in a sealed glass flask.	Aroma associated to strong, piercing, citric smell.	1
Vitamin C	Orange flavoured Vitamin C (*Redoxón*, Argentina)	Related to the aroma of medicine and vitamin complex.	1
Tangerine	5 g of juice powder (“Tangerine”, *Clight*, Argentina) reconstituted in 100 ml of distilled water.	Aromatics related to tangerine juice.	2
Candy	Humidified orange flavoured acid candy (*Sugus,* Argentina*)* placed in a sealed glass flask.	Characteristic odour of orange candy.	3
Solvent	10 g of juice powder (“Tangerine”, *Clight*, Argentina) reconstituted in 50 ml of distilled water.	Aroma associated to solvent and paint.	2
Plastic	Fresh orange juice placed in a plastic recipient, heated for 2 minutes in a microwave oven and stored at room temperature.	Aroma associated to plastic containers.	2
Peely	Filter paper soaked in pure orange essence oil (“Orange peel”, *Ledesma*, Argentina), placed in a sealed glass flask.	Aromatics associated to orange peel	No control could be established.
Marmalade	Orange juice, peel and sugar heated at 85ºC for 60 min.	Aromatics associated to marmalade.	No control could be established.
Woody	Filter paper soaked in orange pure essence oil (“Valencia Orange*”, Ledesma*, Argentina), placed in a sealed glass flask.	Suggestive of the odour of tree bark.	6
Pungency	Orange flavoured, effervescent antacid salts (*Uvasal*, Argentina).	Tingling sensation on the surface of the nose mucosa.	4
Green	No standard was used.	Characteristic odour of unripe orange.	2
Syrup	No standard was used.	Sweet	6

Table 3. Discrimination between samples: triangle test.

Samples compared	Correct answers	Total answers	Significance level
1 / 2	18	24	0.1%
1 / 3	19	24	0.1%
1 / 4	18	24	0.1%
1 / 5	19	24	0.1%
1 / 6	19	24	0.1%
2 / 3	20	24	0.1%
2 / 4	21	24	0.1%
2 / 5	20	24	0.1%
2 / 6	18	24	0.1%
3 / 4	15	24	1.0%
3 / 5	16	24	0.1%
3 / 6	15	24	1.0%
4 / 5	9	24	n.s.
4 / 6	15	24	1.0%
5 / 6	15	24	1.0%

n.s.: no significant difference
Samples 1 through 6 are described in Table 1.

Table 4. F- values of samples for aroma attributes.

Attribute	Anova F-value			
	Replicate (R)	Sample (S)	Assessor (A)	A*S
degrees of freedom	1	5	11	55
Freshly squeezed	0.54	17.99***	0.51	1.96**
Powder juice	0.11	21.37***	1.34	1.30
Citrus terpens	0.57	25.21***	0.95	1.59
Vitamin C	1.28	6.77***	2.99*	1.90*
Tangerine	1.73	3.76**	2.15	1.28
Candy	0.08	10.26***	1.13	0.81
Solvent	2.51	6.78***	0.55	0.98
Plastic	0.92	9.11***	2.37	0.89
Peely	1.79	3.32**	1.58	1.28
Marmalade	3.03	2.69*	0.95	1.44
Woody	0.49	9.77***	0.52	0.98
Pungency	0.44	4.87***	0.98	1.36
Green	0.68	4.98***	2.73	0.83
Syrup	0.06	10.00***	1.42	1.22

*p<0.05; ** p<0.01; ***p<0.001

Table 5. Mean sensory scores of the attributes quantified in each of the samples.

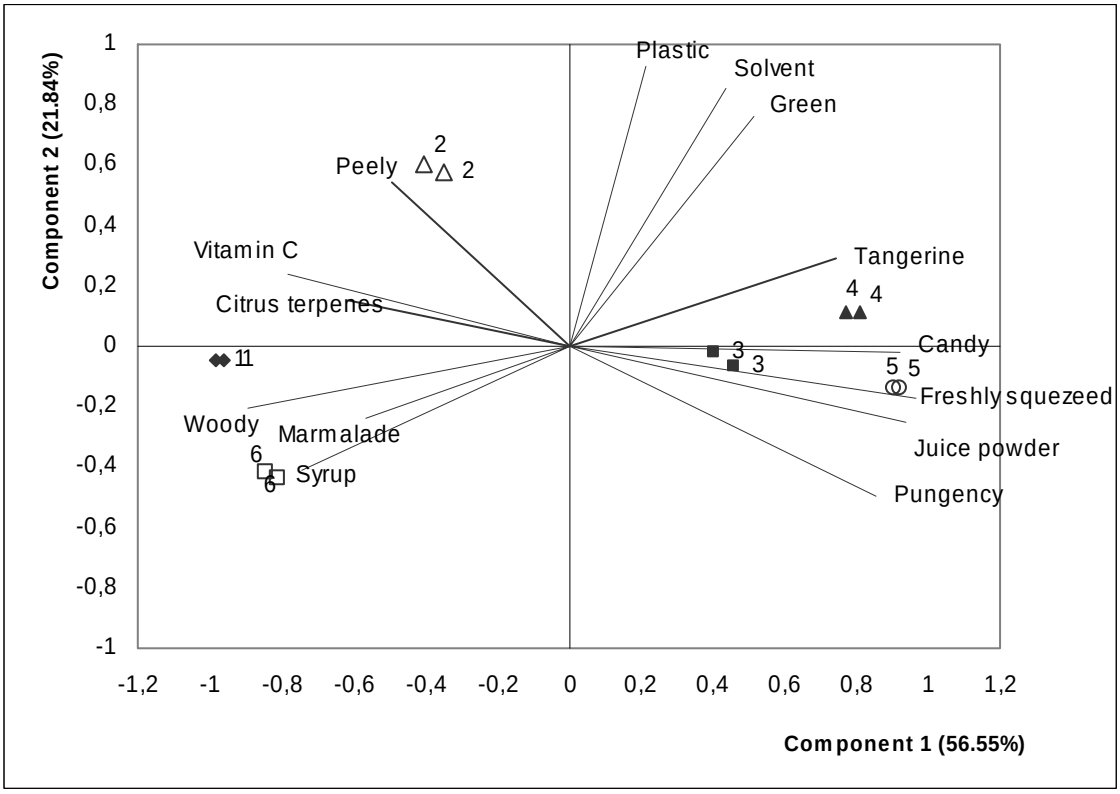
Attribute	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Freshly squeezed	27.6 a	30.4 a	64.6 b	68.9 b	64.3 b	31.4 a
Powder juice	19.2 a	21.9 a	42.5 b	63.8 c	68.4 c	30.0 a
Citrus terpens	75.3 b	35.3 a	23.0a	33.2 a	27.5 a	28.2 a
Vitamin C	65.9 b	46.8 a	38.9 a	31.7 a	31.7 a	38.0 a
Tangerine	31.8 ab	33.3 b	31.7 ab	39.1 bc	45.5 c	20.8 a
Candy	32.6 a	28.7 a	50.8 b	55.6 b	56.5 b	21.0 a
Solvent	31.0 ab	58.5 c	37.5 b	58.6 c	39.5 b	22.0 a
Plastic	29.2 ab	66.9 d	34.2 ab	48.4 c	40.4 bc	23.8 a
Peely	44.6 ab	57.4 b	32.5 a	42.7 ab	28.0 a	41.5 ab
Marmalade	46.4 b	28.5 a	23.3 a	32.7 a	21.7 a	31.4 a
Woody	43.4 b	39.3 ab	30.6 a	26.9 a	30.2 ab	61.4 c
Pungency	26.8 a	25.9 a	39.7 ab	51.4 bc	56.1 c	39.2 ab
Green	31.2 ab	53.5 c	39.9 abc	42.4 bc	49.2 c	27.0 a
Syrup	33.9 ab	44.4 b	36.7 ab	28.6 a	34.1 ab	72.2 c

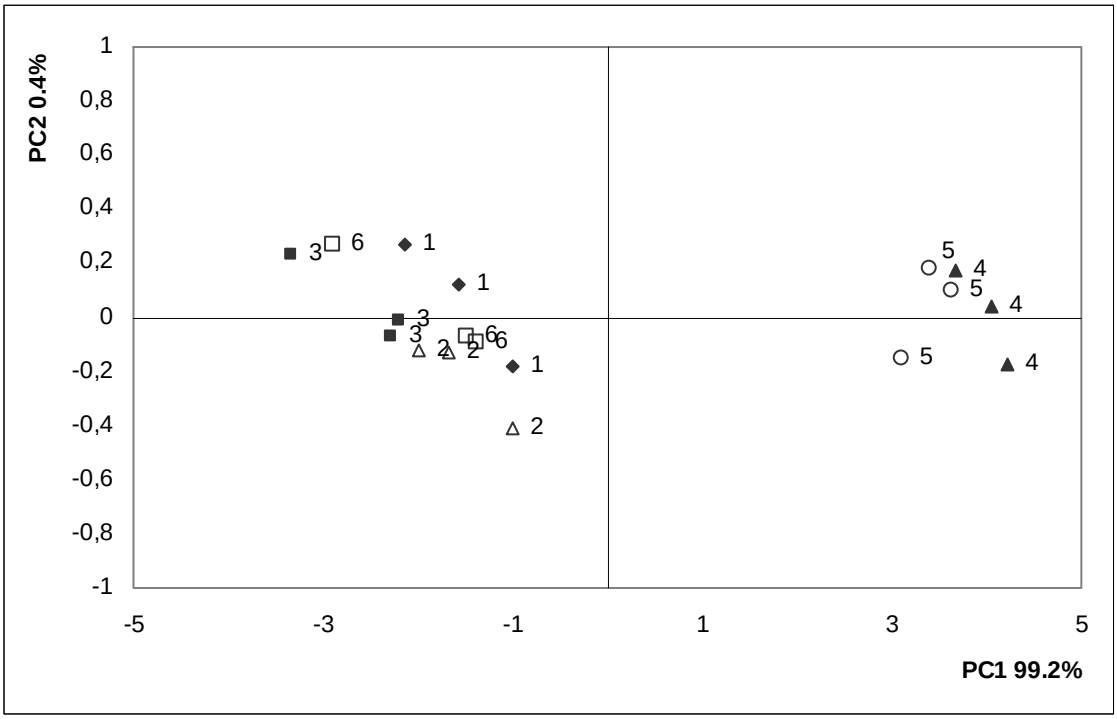
Different letters after medias in every row indicate samples which differed for that attribute, $p<0.05$, SNK test.

Legends for figures

Fig.1. Principal Component Analysis of sensory data.

Fig.2. Principal Component Analysis of e-nose data.




PC2 0.4%
1
0,8
0,6
0,4
0,2
0
-0,2
-0,4
-0,6
-0,8
-1
-5
-3
-1
1
3
5
PC1 99.2%
3
6
1
1
3
3
6
6
2
2
1
2
5
4
5
4
5
4