Sensory evaluation of headspace extracts by direct GC-Olfactometry: application to fresh orange juice

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Abstract

The global sensory quality of solventless extracts of orange juice has been studied by direct GC-Olfactometry (D-GC-O). Headspace of juice and aqueous distillate produced odours closely resembling that of the original juice taken as reference; SPME produced odours with a lower similarity to the reference. Among the SPME fibres used, DVB/CAR/PDMS gave the best sensorial appreciation. Descriptive analysis explained the differences found by similarity scaling. D-GC-O represents a rapid criterion of choice for headspace extraction methods.

Introduction

Orange juice is the most important juice product in the world. Its high quality is greatly dependent on the characteristic "fresh orange juice" flavour produced by a great number of volatile constituents. SPME and headspace techniques are widely used in flavour analysis of orange juice (Jia *et al*., 1998; Moshonas and Shaw, 1997). These techniques have the advantage to be rapid, solventless and cheap. However there are no indications on the sensory properties of the extract. Usually representativeness of extracts is assessed by similarity scaling of extracts incorporated into an appropriate matrix (Etievant *et al.*, 1994). In our case it was not possible to incorporate headspace or SPME extract. This is why we used direct GC-Olfactometry (D-GC-O). This is a new technique which permits to evaluate the global sensory quality of the solventless extracts injected onto a short capillary of deactivated phase directly connected to the GC-sniffing port (Jouquand and Giampaoli, 2000). We tested five different samples obtained from a fresh unpasteurised orange juice: static headspace of the juice, static headspace of an aqueous distillate of the same juice and extracts obtained with three different SPME stationary phases.

Experimental

Fresh orange juice (Naveline, Spain) was obtained by a "Santos" extractor and stored at –30°C under nitrogen atmosphere just until analysis.

Sample extraction

Vacuum distillation (10^{-1} mBar) was performed by the Forss and Holloway apparatus on 1L of juice applying the method described by Guichard and Souty (1988).

For static headspace extraction, aliquots of 5 ml of aqueous distillate and fresh juice were poured into 20 ml vials sealed with PTFE lined caps (Supelco, Bellfonte, PA). Samples were kept 1 h under agitation at 40°C in a water bath. Headspace SPME extraction was performed on 1 ml juice contained in a 4 ml vial sealed with a PTFE lined screw cap. Three different SPME fibres for manual holder were used: 65 μm PDMS/DVB, 75 μm CAR/PDMS, Stableflex 50/30 μm DVB/CAR/PDMS, all the materials were purchased from Supelco (Bellfonte, PA). Prior to SPME, samples were allowed to equilibrate at 40°C under agitation, then volatiles were extracted for 15 min. After adsorption, fibres were retracted and kept ready to D-GC-O analysis.

Direct –GC-Olfactometry (D-GC-O)

A HP 5890 equipped with a sniffing port was supplied with a short capillary of untreated silica (80 cm x 0.32 mm i.d.). The flow rate of the carrier gas (H_2) was 25 ml/min and oven temperature was kept at 50°C. The five extracts were introduced splitless mode into the GC port (T=220°C). For HS samples, 5 ml of the vapour phase at equilibrium were withdrawn by a gastight syringe (Hamilton) and directly injected. SPME fibres were kept into the GC inlet just until the end of the sensorial stimulus. At the sniffing port the effluents were perceived and evaluated by a trained panel of eight assessors.

Sensory analysis

Panellists were first familiarised with five commercial orange juices and asked to agree on a common list of 15 descriptors. A preliminary session was done in order to familiarise the subjects with the D-GC-O device. A similarity test was performed in triplicate on the five samples randomly presented. A time delay of 4 min was maintained between two samples. Sniffers were asked to smell the reference juice contained in a plastic cup sealed with a pierced cap (T=20°C). They had to memorise the odour and then to describe it using the descriptors list. For each stimulus they had to rate its similarity to the reference using a 10 cm scale ranging from 0 (close to the reference) to 10 (far from the reference). They were also asked to give descriptors. Between two olfactory stimuli the assessors had to smell the reference.
Statistical Analyses were realised with the Statbox Software (Grimmer, France). ANOVA and Newman-Keuls test were performed on similarity rates ($p<0.05$); a χ^2 test was effected on descriptive data.

Results and discussion

D-GC-O resulted to be a very rapid and performing tool in sensory analysis of solventless orange juice extracts. The rapidity of the experiment (20 min for each assessor and one repetition) had also the advantage to prevent subjects fatigue. A three ways ANOVA was firstly effected on the similarity rates, taking as possible factors of variation: (1) “method”, (2) “subject ” and (3) “repetition”. Strong “method” and “subject” effects were found ($p<0.001$) with a smaller “method-subject” interaction ($p=0.007$) and no “repetition effect”. Thus, a two ways ANOVA was made on “method” and “subject” effects toward their interaction. The resulting p values indicate that “subject” effect is small in comparison to the “method” effect.

Table 1. Two ways ANOVA for similarity scaling.

	SSE	*DF*	*MS*	*F value*	*P value*
total var.	955.67	118	8.1		
“method” var.	319.02	4	79.75	12.23	0.000007
“subject” var.	200.13	7	28.59	4.39	0.0022
“method*subject” interaction var.	182.52	28	6.52		

Figure 1 shows that the headspace of the juice and of the aqueous distillate produced odours closely resembling that of the original juice taken as reference, with mean scores of 2.64 and 3.44 respectively. These appreciations were found not significantly different by the Newman-Keuls test ($\alpha < 0.05$). In particular, it should be noted that the instrumental device did not produce distortion in odour perception because the headspace of the juice was rated relatively close to the juice reference. On the other hand, SPME extracts gave odours with a poor similarity to reference juice, with scores ranging from 5.20 to 6.95.

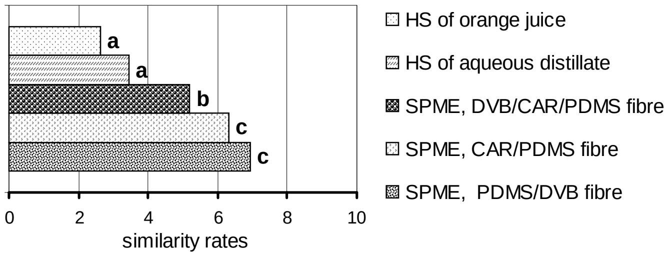

Figure 1. Similarity rates (cm) for the five extracts; same letters indicate means belonging to the same homogenous group (α<0.05).

Among the three different fibres used, the DVB/CAR/PDMS fibre produced the best results. Under the experimental conditions used, it allowed us to obtain a global odour closer to the reference than that obtained by the commonly used PDMS/DVB and CAR/PDMS fibres. This difference is significant at a level of 5%.

In order to explain the differences in sensorial appreciations, we explored descriptive data. Descriptors were grouped in families for a clearer representation and for the χ^2 test. Figure 2 shows the odour profile obtained for each sample and for the reference juice. Descriptive analysis was very coherent with the similarity results and could explain why SPME extracts were rated worst.

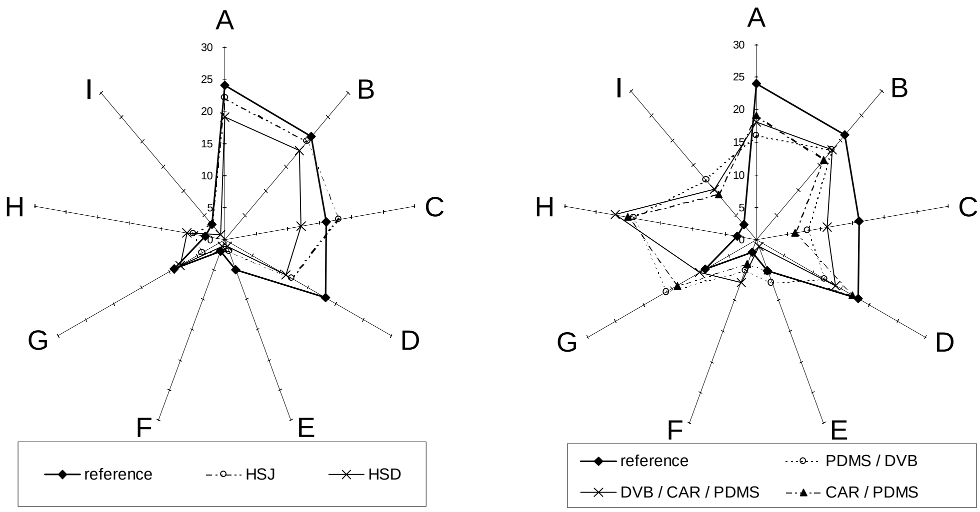

Figure 2. Odour Profiles of the different Headspace extracts: static head space of the juice, HSJ, static headspace of the aqueous distillate, HSD (Left), SPME by the different stationary phases (Right). Alphabetical letters were reported for descriptors. ***A****: orange;* ***B****: orange peel / citrus-like / terpene-like;* ***C****: fresh;* ***D****: fruity;* ***E****: over ripe / cooked;* ***F****: spicy / woody;* ***G****: sweet / caramel-like / vanilla-like;* ***H****: metallic / oxidised / plastic / waxy / smoky / mouldy;* ***I****: artificial / chemical.*

Headspace of the juice (HSJ) and of the distillate (HSD) gives sensorial profiles very similar to the reference one, with predominant “orange” (**A**), “fresh” (**C**), “citrus” (**B**) and “fruity” (**D**) notes. In SPME odour profiles pleasant notes (**A** to **D**) were perceived, however unpleasant notes appeared, gathered in group **H**: “metallic, oxidised, plastic, waxy, smoky and mouldy”. Assessors also perceived some chemical (**I**) and caramel-like (**G**) notes higher than in HS samples. A χ^2 test effected on these group of descriptors showed a good correlation (p=0.00054) between descriptors and extracts. In particular a strong contribution to the test was given by the couple of variables “HSJ” and “fresh” (**C**) and also by unpleasant attributes (**H**,**I**) and all the SPME extracts.

SPME is largely applied in the analysis of orange juice and it is often coupled to GC-O (Bezman *et al*., 2001). Many works are done on the optimisation of extraction conditions, namely the type of solid phase, the time and the temperature of extraction (Bazemore *et al*., 1999). D-GC-O showed that even if SPME conditions were accurately chosen, they gave extracts with low similarity. SPME could probably cause a concentration effect in the molecules responsible for unpleasant notes which are not due to SPME fibre. Even if SPME permits to efficiently extract volatile compounds, an imbalance of odour active compounds could be obtained. D-GC-O allowed us not only to evaluate SPME extracts but also to discriminate among different fibres. Globally, the three fibres gave similar sensory profiles. Nevertheless the “over ripe / cooked” (**E**) and the “caramel-like” (**G**) notes are less pronounced for the DVB/CAR/PDMS fibre extract which resulted also more “fresh” (**C**) than the others. These descriptive results could explain the better performance of DVB/CAR/PDMS fibre in similarity rates. “Classical” GC-Olfactometry and quantitative analysis of the volatile compounds present in the different extracts is going on in order to understand which differences in key aroma compounds are responsible for the different global perception.

Conclusion

D-GC-O is a rapid and performing tool in sensory analysis of headspace extracts. We developed a simple methodology to evaluate the representativeness of orange juice extracts. The high odour similarity found for the juice headspace is a validation to this method. We showed that, using standard experimental conditions, poorly resembling extracts are obtained. Moreover, D-GC-O allowed to sensorially discriminate different SPME fibres. D-GC-O, thus, could be used to choose the solid phase and extraction conditions which give the odour most representative of the original juice.

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