doi: 10.17221/303/2015-CJFS

Detection of Adulteration in Freshly Squeezed Orange Juice by Electronic Nose and Infrared Spectroscopy

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Abstract:

Shen F., Wu Q., Su A., Tang P., Shao X., Liu B. (2016): **Detection of adulteration in freshly squeezed orange juice by electronic nose and infrared spectroscopy**. Czech J. Food Sci., 34: 224–232.

The use of electronic nose and attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) as rapid tools for detection of orange juice adulteration has been preliminarily investigated and compared. Freshly squeezed orange juices were tentatively adulterated with 100% concentrated orange juices at levels ranging from 0% to 30% (v/v). Then the E-nose response signals and FTIR spectra collected from samples were subjected to multivariate analysis by principal component analysis (PCA) and linear discriminant analysis (LDA). PCA indicated that authentic juices and adulterated ones could be approximately separated. For the classification of samples with different adulteration levels, the overall accuracy obtained by LDA in prediction was 91.7 and 87.5% for E-nose and ATR-FTIR, respectively. Gas chromatography-mass spectrometry (GC-MS) results verified that there existed an obvious holistic difference in flavour characteristics between fresh squeezed and concentrated juices. These results demonstrated that both E-nose and FTIR might be used as rapid screening techniques for the detection of this type of juice adulteration.

Keywords: chemometrics; discrimination; spectral analysis; volatile compounds

Adulteration of juice products for economic benefits is a well-established malpractice and has become a serious economic problem for centuries (Manning & Soon 2014). Nowadays, juice adulteration has progressed from simple dilution with water and sugar to more sophisticated manipulations, such as addition of a less expensive fruit juice that is designed to mask the illicit process (Twomey *et al.* 1995). Freshly squeezed fruit juices, claimed to be 100% natural, and not from concentrate (NFC), are only those juices that are produced exclusively from the fleshy part of fruit, with no water, sugars and other added ingredients. Freshly squeezed juices are considered not to be processed by means of any novel techniques such as HPP (High Pressure Pasteurisation) which preserves the whole freshness of juices and their organoleptic and nutritional characteristics (Hong & Wang 2014). The flavour and compositions of fresh juice can be easily changed by heat or pressure treatment during processing. Nisperos-Carriedo and Shaw (1990) demonstrated that pasteurised reconstituted orange juices from concentrate showed remarkable differences in flavour characteristics as compared to fresh juices. Changes in carotenoid pigment content and juice colour due to thermal pasteurisation were also verified in Valencia orange juices (Lee & Coates 2003). Velázquez-Estrada *et al.* (2013) revealed significant differences in some bioactive compounds (such as flavanone content) of fresh squeezed orange juice that undergone ultra-high pressure homogenisation (UHPH) treatments. All these alterations may have negative impacts on the

Supported by the National Natural Science Foundation of China, Grant No. 31301482, National Key Technology Support Program, Project No. 2015BAD19B03, Special Fund for Grain Scientific Research in the Public Interest, Grant No. 201513002-5, and by the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD).

doi: 10.17221/303/2015-CJFS

taste and nutrition of fresh juice. Thus, fresh juices have gained a high reputation for health benefits and consequently are a growing market in modern societies (Faria *et al.* 2013). However, owing to relative high price as well as short storage period of fresh juices, adulteration of fresh juices with cheaper or processed juices seems to be happening. Hence authentication of freshly squeezed juice products is of crucial importance both from consumer and regulatory perspectives.

Traditionally, distinguishing between authentic and adulterate fruit juices has often been carried out by sensory evaluation or chromatographic techniques, such as high performance liquid chromatography (HPLC), gas chromatography-mass spectrometry (GC-MS), and ion chromatography (Bleibaum *et al*. 2002; Gómez-Ariza *et al*. 2005; Reinhard *et al*. 2008; Guyon *et al*. 2013). The chemical compositions of juices from one specific chemical class (i.e., sugars, flavonoids, organic acids, etc.) could be determined for comparison and identification purposes (Saavedra *et al.* 2000). However, they often suffer from some disadvantages, namely time-consuming, expensive, and requiring significant expertise or skilled personnel, which limit their applications to rapid detection and process control (Huang *et al.* 2015). Therefore, there is a trend to develop rapid, simple, efficient, and on-site analysis methods for the quality inspection of juice products.

Recently, electronic nose (E-nose) and Fourier transform infrared spectroscopy (FTIR) techniques have been gradually developed as alternatives to wet chemistry in the food industry and agriculture, mainly because they can be applied in a low-cost, rapid, and non-destructive way (Mamat *et al*. 2011; Shen *et al*. 2011; Králová *et al.* 2014). E-nose comprises a series of gas sensors which have sensitivity and selectivity to volatile compounds present in the sample headspace of samples. It can provide an overall estimate of the volatiles present in a sample rather than any specific information. FTIR technique, a well-established technique in chemical analysis, is based on the measurement of the frequencies of chemical bonds in functional groups such as C-C, C-H, O-H, C=O, and N-H, upon absorption of radiation in the mid-infrared region (400–4000 cm^{-1}) (Tena *et al*. 2013). In addition, attenuated total reflectance (ATR) measurements for FTIR offer interesting possibilities for the analysis of samples containing solids and liquids (Beullens *et al.* 2006). In the combination of multivariate analysis, the potential of E-nose and FTIR techniques is greatly expanded. Currently, various studies have applied E-nose and IR to the analyses of melons (Sánchez *et al*. 2014), peaches (Hui *et al.* 2012), and various fruit juices for quality control (Haddi *et al*. 2014), fresh and shelf-life evaluation (Egidio *et al*. 2009), process monitoring (Sinelli *et al*. 2011) as well as authenticity assessment (Zielinski *et al.* 2014).

However, in terms of adulteration manipulations, little attention was paid to the addition of 100% concentrated juices into freshly squeezed juices, which could be a more sophisticated situation for detection because of almost the same chemical compositions and flavour. Orange juice is probably the best known and most widespread fruit juice all over the world, particularly appreciated for its flavour and high beneficial value. Therefore the main objective of this study was to investigate the application of E-nose and FTIR techniques to differentiate freshly squeezed orange juices and the ones adulterated with concentrated orange juices at different levels (10–30%).

MATERIAL AND METHODS

Samples. Freshly squeezed orange juice samples of one brand (Ling du guo fang) and 100% concentrated orange juice samples of three brands (Weiquan, Dahu, and Huiyuan) were all purchased from large regular supermarkets in Nanjing. The authenticity of the samples was approved and permitted by the local governmental food regulatory agencies. No significant difference in their appearance could be observed by the naked eyes. The freshly squeezed juice samples were then mixed in proportions ranging from 10% to 30% of concentrated juices for each adulteration level, in 10% increments (v/v). Samples with different adulteration levels were prepared, which were 0, 10, 20, and 30% (by volume), respectively. Totally, 18 control freshly squeezed juice samples (6 lots × 3 replicates), and 54 adulterated samples (3 adulteration levels × 6 replicates × 3 brands) were obtained for further studies. Before blending, all juices were filtered using medical gauze that was folded into four layers. After blending, all samples were shaken for 30 min on a shaking table and stored under refrigerated (0–4°C) conditions until analysis.

E-nose measurement. The headspace analysis of juice samples was performed with a FOX 3000 E-nose (Alpha MOS, Toulouse, France). It is equipped with 12 metal oxide gas sensors of different selectivity

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and sensitivity towards volatile compounds. The FOX 3000 E-nose system consists of a sampling apparatus, a detector unit containing the array of sensors and pattern recognition software for data recording and analysis. Five ml of orange juice was placed in a 20 ml bottle for E-nose analysis to provide a vapour phase in equilibrium with the liquid. Two replicates for each sample were carried out. The characteristics of the sensors used in E-nose as well as the optimal operating conditions could be found elsewhere (Hartyáni *et al.* 2013).

ATR-FTIR measurement. (ATR-FTIR) spectra of samples were obtained using a commercial FTIR spectrometer (Bruker Tensor 27; Bruker Optik, Ettlingen, Germany), which was equipped with an interferometer and a deuterated triglycine sulphate (DTGS) detector with KBr beamsplitter. The equipment was combined with a horizontal attenuated total reflectance (HATR) accessory with a ZnSe crystal (Pike Technologies, Madison, USA) for ATR measurement. The measurements were directly carried out by dripping 0.5 ml juice samples on the ATR surface at controlled room temperature (20°C) in a region of 800–4000 cm^{-1}, by accumulating 32 scans with a resolution of 4 cm^{-1}. Sample spectra were scanned in triplicate and displayed as the average spectra. A reference spectrum of air was acquired before the measurement of each juice sample replication. Ultrapure water was used as a reference sample. At the end of each scan, the ATR crystal was cleared with deionised water and then wiped dry using a soft tissue paper to avoid cross-contamination.

Headspace solid phase micro-extraction and GC-MS. The volatile compounds of fruit juices were analysed by headspace solid phase micro-extraction (HS-SPME) and gas chromatography-mass spectrometry (GC-MS). An SPME device (Supelco, Bellefonte, USA) with a 10 mm fibre coated with 50/30 µm DVB/CAR/PDMS was used for the extraction. After extraction, the SPME device was introduced in a GC splitless injector and maintained at 250°C for 5 minutes. Measurements were then carried out using an Agilent 7890A GC coupled with an Agilent 5975C mass selective detector. The separation was carried out by an HP-5MS capillary column (30 m × 0.25 mm, 0.25 µm). Helium was used as a carrier gas. The injector and detector temperature was 250 and 280°C, respectively. The oven temperature chromatographic program was set at 50°C initially, raised at 8°C/min to 125°C (held for 3 min), then raised at 4°C/min to 165°C (held for 3 min), at last raised at 10°C/min to 230°C (held for 2 min). The peaks were identified by comparing their mass spectra with the NIST library (NIST 2008), and compounds with a > 80% match were used by referring to other authors (Stoppacher *et al*. 2010).

Multivariate statistical analysis. All the data analysis was carried out using Matlab software (Version 7.0; The MathWorks Inc., Natick, USA). Principal component analysis (PCA) was first applied to display any possible patterns and outliers between samples. PCA can transform original variables into a few new ones, known as principal components (PCs). The PCs account as much as possible for the variability in the original variables and they are orthogonal to each other to remove overlapped information in the original spectra matrix (Yu *et al.* 2006). Discrimination models were then developed by linear discriminant analysis (LDA). LDA is a supervised technique that is widely recognised in classification problems. It is based on the determination of linear discriminant functions, which maximises the ratio of between-class variances by minimising the within-class variances. The discriminant function is called the canonical variable. It is obtained in the way that it produces a new latent variable, which is a linear combination of the original variables (Xie *et al*. 2010).

RESULTS AND DISCUSSION

E-nose response to orange juices. The typical responses of 12 E-nose sensors to an authentic sample and an adulterated sample are shown in Figure 1. The *x*-axis represents time, and the *y*-axis represents intensity (G/G_0), where G and G_0 stand for the conductivity of the sensor when the sample gas and the zero gas blow over, respectively. Each curve represents the change of sensor conductivity during measurement. The conductivity of each sensor gradually changed (increased or decreased) during 1–30 s and finally reached a dynamic balance at approximately 60 s. Sensors of LY2/GH, LY2/gCTL, and P10/2 were found to be the most sensitive to juice samples, mainly related to aldehydes in samples. A significant change of LY2/AA for authentic sample could be observed compared to adulterated sample, which was considered to be more useful and helpful for identification of adulteration. Meanwhile, LY2/gCT did not change so much conspicuously as other sensors did. In order to reduce the dimension of the data matrix, by referring to other authors, only the

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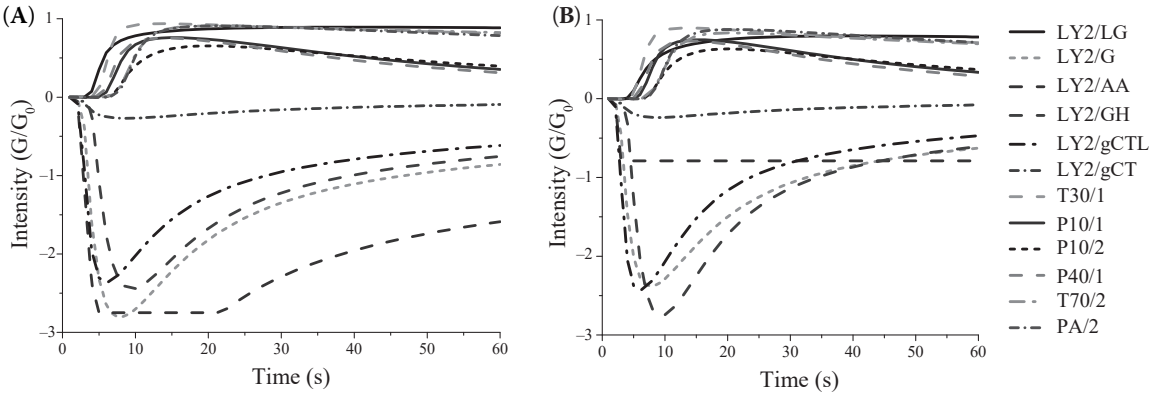

Figure 1. E-nose response curves of 12 sensors to an authentic juice sample (**A**) and an adulterated sample (**B**)
G – conductivity of the sensor when the sample gas blow over; G_0 – conductivity of the sensor when the zero gas blow over

maximum response values of the 12 sensors were extracted for each sample in further statistical analysis (Trirongjitmoah *et al*. 2015).

ATR-FTIR spectra of samples. ATR-FTIR spectra of juice samples with different adulteration levels are shown in Figure 2, which are dominated by water and sugar absorptions. The spectrum of ultrapure water was also collected to make a comparison. The main absorption at 3200–3400 and 1640 cm^{-1} is mainly related to O-H vibrational group in water. The main difference between ultrapure water and orange juice could be found at 1500–950 cm^{-1}. Sugars and organic acids also show intense and characteristic bands in this region. Specifically, the bands in the region of 1500–1000 cm^{-1} arise mainly from the deformation of $-CH_2$ and angular deformation of C-C-H and H-C-O (Shiroma & Rodriguez-Saona 2009). Those in the region of 1200–950 cm^{-1} can be explained by stretching modes of C-C and C-O (Ferreira *et al*. 2014).

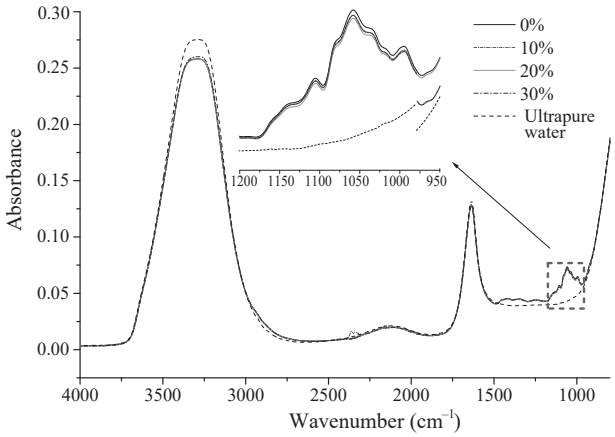

Figure 2. Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) spectra of samples with different adulteration levels

However, the four spectra were highly overlapped, and no obvious differences were observed between them from visual inspection. It could be deduced that the distinction between authentic and adulterated samples in chemical compositions was small. After close examination, slight differences could be observed between samples from different groups in the range of 1200 and 950 cm^{-1}. The absorption was generally decreased with the increase of adulteration levels, which might provide a basis for discrimination by ATR-FTIR spectroscopy.

GC-MS results. GC-MS results indicated that more than 70 volatile compounds have been identified in fresh and adulterated orange juices. Among them, the average peak area (%) of 19 major volatile compounds belonging to alkenes, alcohols, aldehydes, ketones, and esters is summarised in Table 1, which were found to be identical in category but different in abundances. As reported in the literature, most terpenes, including D-limonene, β-myrcene, α-pinene, and α-terpinene, were higher in freshly squeezed juices (Johnson *et al*. 1996). Because of the ultrafiltration and evaporation during the concentration process, the predominant volatile flavour is almost entirely stripped from fresh juice (Johnson & Vora 1983). However, the relative content of α-terpineol and carvone was higher in concentrated juices, which was related to the oxygenolysis of D-limonene during the processing (Pierce *et al*. 1996). Citral, decanal, ethyl butanoate, and ethyl 3-hydroxyhexanoate were considered as important contributors to the citrus aroma, as well as a slight decrease in concentrated juices was similar to the results in the literature (Bai *et al*. 2010). Moreover, minor changes of other volatiles could also be observed between freshly squeezed and concentrated juices. The results suggested that the

doi: 10.17221/303/2015-CJFS

Table 1. Average area percentage of key volatile compounds in freshly squeezed and concentrated orange juices obtained by GC-MS

Volatile compound	Peak area (%)			
	Ling du guo fang	Weiquan	Huiyuan	Dahu
Alkenes				
D-Limonene	72.47	48.96	53.40	48.44
α-Pinene	0.62	0.42	0.31	0.28
β-Myrcene	1.89	0.88	1.19	0.87
4-Carene	0.10	0.11	0.07	0.16
Caryophyllene	0.34	0.35	0.44	0.27
α-Terpinene	0.55	0.14	0.39	0.27
Copaene	0.26	0.22	0.39	0.27
Alcohols				
Linalool	1.43	1.58	2.11	1.64
α-Terpineol	1.54	5.48	4.21	4.15
1-Octanol	0.11	0.25	0.18	0.28
Aldehydes				
Citral	0.27	0.11	0.24	0.15
Nonanal	0.27	0.31	0.28	0.13
Decanal	0.68	0.31	0.55	0.48
Acetaldehyde	0.12	0.11	0.23	0.18
Ketones				
Carvone	0.34	0.52	0.68	0.53
Esters				
Ethyl butanoate	1.35	1.08	1.11	1.13
Ethyl 3-hydroxy-hexanoate	0.41	0.26	0.11	0.14

flavour of fresh juice might exhibit unique flavour characteristics as compared to concentrated juices.

PCA results. PCA was firstly performed to reveal any possible groupings of the samples. The potential of both E-nose and ATR-FTIR to classify adulteration levels based on their response signal was evaluated. The first PCs of the PCA performed on the E-nose data explained 97.9% of the variance. A general separation between freshly squeezed juice samples and adulterated ones could be observed (Figure 3A). Only several samples were mixed with each other, which indicated that there existed holistic differences in volatile compounds, in accordance with GC-MS results. However, the separation between samples of different adulteration levels (10, 20, and 30%) was not satisfactory. This deficiency could be partly assigned to the fact that the intra-flavour variability between fresh and concentrated juices was quite small. The score plot of PC1 and PC2 derived from ATR-FTIR spectra is also shown in Figure 3B. Similarly, an approximate separation trend could be observed between authentic samples and adulterated ones. The discrimination between samples of different adulteration levels could not be found either. The results might suggest that the chemical compositions between samples were quite similar. However, the classification obtained by PCA is not perfect. PCA only indicates the visualising of dimension spaces. Therefore, discriminant analysis is used for an improved separation.

LDA results. In this step, LDA was applied to build calibration models to classify juice samples into four adulteration levels (0–30%). In order to cover

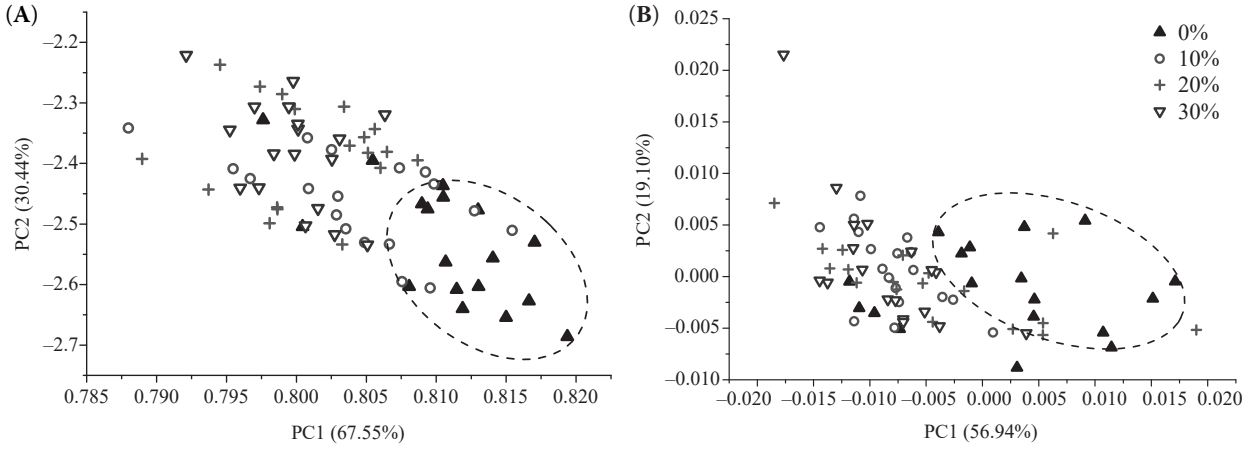

Figure 3. The score plot of PC1 and PC2 of freshly squeezed juice and adulterated juice samples resulting from (**A**) E-nose and (**B**) attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR)

doi: 10.17221/303/2015-CJFS

Table 2. Classification results of juice samples with different adulteration levels obtained by linear discriminant analysis (LDA) based on E-nose and and attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR)

Adulteration level group	LDA based on E-nose					LDA based on ATR-FTIR				
	predicted group membership				accuracy (%)	predicted group membership				accuracy (%)
	0%	10%	20%	30%		0%	10%	20%	30%	
Calibration										
0%	11	1	0	0	91.67	11	1	0	0	91.67
10%	0	12	0	0	100	1	11	0	0	91.67
20%	0	0	12	0	100	0	0	12	0	100
30%	0	0	0	12	100	0	0	2	10	83.33
Total					97.92					91.67
Validation										
0%	4	2	0	0	66.67	6	0	0	0	100
10%	0	6	0	0	100	1	5	0	0	83.33
20%	0	0	6	0	100	0	0	6	0	100
30%	0	0	0	6	100	2	0	0	4	66.67
Total					91.67					87.50

most of the variance contained in the raw data, the maximum response values of the 12 E-nose sensors and the first 10 PCs extracted from MIR spectra for each sample were included in an LDA model, respectively. 48 samples were randomly selected as the calibration set, while the remaining 24 samples were separated as the validation set.

Table 2 shows the classification results obtained by E-nose. The model had an overall correct classification rate of 97.9% and a validation rate of 91.7%. In calibration, only one sample from the authentic group was misclassified. Meanwhile, 2 samples from the authentic group were misclassified, which resulted in a correct classification rate of 66.7% in its own group. However, the discrimination between samples from adulteration levels of 10, 20, and 30% was satisfactory with classification accuracy of 100%. The LDA scatter plot of two discrimination functions regarding samples with various adulteration levels is given in Figure 4A. A much better result was obtained than that

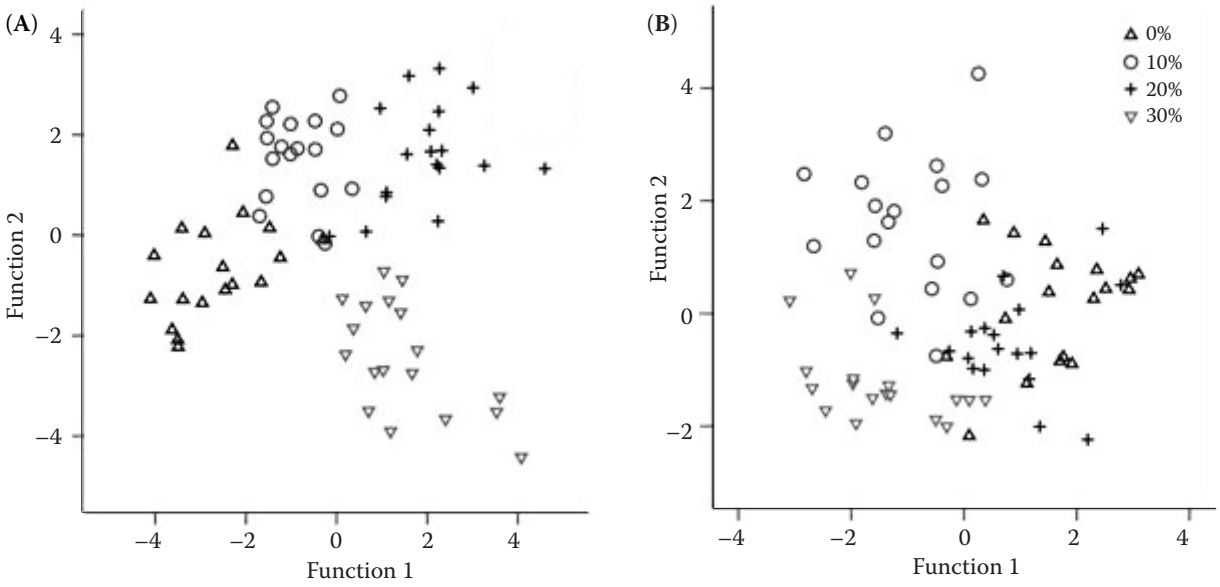

Figure 4. The LDA scatter plot of two discrimination functions regarding samples with different adulteration levels resulting from (**A**) E-nose and (**B**) attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR)

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of PCA. It can be explained that LDA is a supervised method. Differences between samples are magnified from each other. It could be seen that except for some individual samples overlapped, the four groups were clearly separated, which indicated that LDA coupled with E-nose data was very sensitive to the subtle difference in flavour characteristics between samples.

The discrimination results obtained by ATR-FTIR are shown in Table 2. Discrimination between samples was a little less pronounced than that by E-nose. Four samples were misclassified, which resulted in a correct classification rate of 91.7% in calibration. In validation, 3 samples were misclassified and the classification accuracy in validation was 87.5%. The LDA score plot is also shown in Figure 4B. Better performance was obtained compared to that of PCA, except for some samples from the 20% adulteration level that were mixed with other groups, while the remaining samples were basically separated. In summary, although the differences in the chemical composition and flavour between samples might be quite small, the LDA method gave an acceptable degree of accuracy in prediction (> 85.0%). The results suggested that both ATR-FTIR and electronic nose had the feasibility as a quick method for detection of this type of adulteration.

In general, GC-MS results demonstrated that some volatile compounds, such as D-limonene, β-myrcene, α-pinene, α-terpinene, α-terpineol, carvone, citral, decanal, ethyl butanoate, and ethyl 3-hydroxyhexanoate, might be very important for discrimination between fresh and concentrated juice. As reported in the literature (Reinhard *et al*. 2008), the GC-MS method was relatively reliable and sensitive for juice classification, which presented better results than the E-nose technique. However, its operation and data handling were more complicated and time consuming. E-nose could reveal the information about changes in volatiles from juices, and the spectral features associated with organic acids, sugars, and esters in samples could be obtained by the FTIR method. It is probable that freshly squeezed juices undergo changes in their flavour and chemical compositions during processing such as high pressure pasteurisation. These changes could be reflected in their responses of E-nose sensors as well as FTIR spectra, which made both methods viable. Nevertheless, it was hard to define a single compound or several compounds that can explain the classification between juices due to the nature of the chemometric methods used (Liu *et al*. 2006). The information about many chemical compounds contained in orange juices recorded by E-nose and ATR-FTIR can act as "fingerprints" for the discrimination of juices.

CONCLUSIONS

In conclusion, the results demonstrate that both E-nose and ATR-FTIR techniques combined with multivariate analysis are able to discriminate freshly squeezed orange juices adulterated by concentrated juices. In this study, the prediction accuracy obtained by the two methods was 91.7 and 87.5%, respectively. Such a methodology provides a rapid, low-cost tool for identification of juice origin and process control. However, some factors limit the applicability of the classification models, such as the number of samples used to develop the calibration model and the similarities between samples. Therefore, before the method can be used by the juice industry with confidence, more samples from different origins, varieties and brands should be incorporated to build a more robust model.

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Received: 2015–06–15
Accepted after corrections: 2016–04–28
Published online: 2016–06–01

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