

Zheng Hai  
Jun Wang

Department of Agricultural  
Engineering, Zhejiang University,  
Hangzhou, P. R. China

## Detection of adulteration in camellia seed oil and sesame oil using an electronic nose

An electronic nose was used for the detection of maize oil adulteration in camellia seed oil and sesame oil. The results of multivariate analysis of variance showed that the sensor signals of different kinds of oil are significantly different from each other. Principal component analysis (PCA) cannot be used to discriminate the adulteration of camellia seed oil, but can be used in the discrimination of adulteration in sesame oil. Linear discriminant analysis (LDA) is more effective than PCA and can be used in adulteration discrimination for both camellia seed oil and sesame oil. In order to check the discriminative power of LDA, canonical discriminant analysis was performed as well. Acceptable results were also obtained: The accuracy of prediction was 83.6% for camellia seed oil and 94.5% for sesame oil. The artificial neural network (ANN) model was used to detect the percentage of adulteration in camellia seed oil and sesame oil. The results showed that, based on ANN as its pattern recognition technique, the electronic nose cannot predict the percentage of adulteration in camellia seed oil, but can be used in the quantitative determination of adulteration in sesame oil.

**Keywords:** Camellia seed oil, sesame oil, adulteration, electronic nose.

### 1 Introduction

Camellia seed oil is extracted from the seed of camellia and abounds in unsaturated fatty acids, so it is very nutritional and popular in many Asian countries. Sesame oil contains a multiplicity of compounds with potentially beneficial biological activities, such as antioxidant and cardio-protective properties [1], and has a full-bodied odor and a pleasant taste and, as such, is a natural salad oil requiring little or no winterization. These two kinds of oil are all more expensive than other kinds of edible vegetable oil. Therefore, adulterating them with other cheaper or lower-quality oil could be very lucrative [2]. Although in most cases adulteration does not pose a threat to public health, fundamental rights of consumers are violated by fraudulent malpractice [3]. The most frequent adulterations are those carried out with sunflower oil, maize oil and bean oil.

Most of the current work on edible oil adulteration is based on chromatographic analysis and chemical analysis. These methods are all time consuming and tedious [4–10]. Thus, application and development of new methods to detect adulteration in vegetable oils are of prominent importance in order to vindicate consumers' rights.

Among these approaches are infrared spectroscopy [11], mass spectrometry [12, 13] and nuclear magnetic resonance [14]. Another reported technique for oil authentication involves measurement of stable carbon isotope ratios. However, most of these techniques are usually time consuming and costly for routine use in the food industry; there is a large demand for rapid, cheap and effective techniques for quality control of food products.

The concept of an electronic nose (E-nose) was proposed in 1982 at the University of Warwick by Persaud and Dodd. It comprises several kinds of hardware such as sensors, electronics, pumps, air conditioner, flow controller, etc., and is coupled with a pattern recognition technique. 'Electronic nose' systems were designed to be used with numerous products, such as cars, food, packaging, cosmetics, etc. As far as food is concerned, many investigations have been reported, such as differentiation and classification in meat, coffee, cheese, beer, grain, fish, fruit and others [15–22]. As for oil, several scholars have reported that, based on principal component analysis (PCA), linear discriminant analysis (LDA) or artificial neural networks (ANN) as pattern recognition techniques, electronic noses can discriminate different kinds of vegetable oil from each other [23–30]. Gan reported that the electronic nose based on acoustic wave (SAW) sensors can discriminate fresh oil from rancid oil and may be utilized as an analytical tool to follow the progress of oxidation and breakdown of vegetable oil [31]. Ma's experiment indicated that, based on LDA, the

**Correspondence:** Jun Wang, Department of Agricultural Engineering, Zhejiang University, 268 Kaixuan Road, Hangzhou 310029, P.R. China. Phone: +86 571 86971881, Fax: +86 571 86971139, e-mail: jwang@zju.edu.cn

electronic nose can differentiate adulterated and non-adulterated olive oil from each other very well [2]. Che Man demonstrated that it was possible to detect lard adulteration (as low as 1%) in refined, bleached and deodorized palm olein using the surface acoustic wave sensing electronic nose [32]. In our work, the attempt was to discriminate three kinds of oil, maize oil, camellia seed oil and sesame oil, and to detect maize oil adulteration in sesame oil and camellia oil with an electronic nose based on ten metal oxide semiconductor sensors.

## 2 Materials and methods

### 2.1 Samples

Refined camellia seed oil, sesame oil and maize oil were purchased from a local super market. Before examination, samples were stored in the dark and none of them was subjected to any treatment that might alter their composition.

### 2.2 Blend preparations

The three pure oils (camellia seed, sesame oil and maize oil) were prepared. The camellia seed oil (or sesame oil) was mixed in proportions ranging from 10 to 90% of maize oil for each adulteration level, in 10% increments (vol/vol); nine groups of samples with different adulteration levels were prepared, which were 10, 20, 30, 40, 50, 60, 70, 80 and 90% (by volume) adulterated by maize oil, respectively.

For each group, 15 samples were carried out and detected by the E-nose. So there were 315 samples for further studies. Of these, 45 samples were three kinds of pure oils and 270 samples were blends (135 samples for each mixed sesame oil or each mixed camellia seed oil). For each sample, 20 mL pure oil or blend was injected into a container and airtight for 1 h before measuring with the electronic nose.

### 2.3 Electronic nose apparatus

An Airsense M.O.S. Electronic Nose System, the PEN2, was used to obtain the chemical vapor prints of the samples. It has ten metal oxide gas sensors covering a broad range of molecules that can be present in the headspace of the samples. The nomenclature and characteristics of the sensors used are shown in the Tab. 1, and the operation temperature of the sensors was 300 °C.

The sample gas was sucked into the sensor chamber through the inlet at a rate of 400 mL/min. Zero gas was pumped from its port at the backside of the instrument into the sample gas path. During the flushing mode, this flow rate was adjusted to 600 mL/min so that the sample line connected to the inlet was rinsed backwards at 200 mL/min. In order to achieve a safe identification from the measurement, PEN includes the 3A technology: automatic ranging, automatic calibration and automatic enrichment.

**Tab. 1.** Sensors used and their main applications in PEN2.

Number in Array	Sensor Name	General Description	Reference
1	W1C	Aromatic compounds	Toluene, 10 ppm
2	W5S	Very sensitive, broad-range sensitivity, reacts to nitrogen oxides, very sensitive with negative signal	NO <sub>2</sub> , 1 ppm
3	W3C	Ammonia, used as sensor for aromatic compounds	Benzene, 10 ppm
4	W6S	Mainly hydrogen, selective, (breath gases)	H <sub>2</sub> , 100 ppb
5	W5C	Alkanes, aromatic compounds, less polar compounds	Propane, 1 ppm
6	W1S	Sensitive to methane (environment) ca. 10 ppm. Broad range, similar to No. 8	CH <sub>4</sub> , 100 ppm
7	W1W	Reacts to sulfur compounds, H <sub>2</sub> S 0.1 ppm. Otherwise sensitive to many terpenes and sulfur organic compounds, which are important for smell, limonene, pyrazine	H <sub>2</sub> S, 1 ppm
8	W2S	Detects alcohols, partially aromatic compounds, broad range	CO, 100 ppm
9	W2W	Aromatic compounds, sulfur organic compounds	H <sub>2</sub> S, 1 ppm
10	W3S	Reacts to high concentrations >100 ppm, sometimes very selective (methane)	CH <sub>4</sub> , 10 CH <sub>4</sub> , 100 ppm

## 2.4 Electronic nose measurement

In recent years, techniques based on the generation of a headspace have been developed. These are particularly attractive in the sense that they measure volatile substances in the same way as the human olfactory system works. The dynamic headspace sampler, which has the advantage of including a pre-concentration step, thereby improving detection limits, was employed. In order to improve the robustness of the experiments, the samples were injected in a random order.

After a headspace generation time of 1 h, the volatile compounds generated were pumped at a speed of 400 mL/min through the measurement chamber containing the array of gas sensors. During the headspace generation time, the stream of zero gas was blown into the measuring chamber to enable the gas sensor signal to return to baseline (approximate desorption time 100 s). Upon injecting the sample, data were acquired every second over 60 s.

Fig. 1 shows typical recordings for sesame oil, maize oil and camellia seed oil samples. The signals corresponding to sesame oil are greatly different from those of camellia seed oil and maize oil. For example, when the E-nose was exposed to the volatiles of sesame oil, the ratio of conductivity ( $G/G_0$ ) expanded to more than 10 in 15 s, which is much greater than the ratios after exposure to the volatiles of camellia seed oil and maize oil. When the sensors were exposed to the volatiles of camellia seed oil or bean oil, the ratio of conductivity ( $G/G_0$ ) changed more evenly. The recordings of camellia seed oil and maize oil are subtly different from each other.

## 2.5 Data analysis

### 2.5.1 PCA

PCA was used to achieve a reduction of dimension and to observe a primary evaluation of the between-class similarity. PCA is a projection method that allows an easy visualization of all the information contained in a data set. In addition, PCA helps to find out in what respect one sample is different from another and which variables contribute most to this difference. This pattern recognition technique is used in order to observe similarities among different oil samples, reducing the dimension from ten variables to two or three principal components and keeping most of the original information content in the data set. This pattern recognition analysis was performed by means of the WinMuster 1.6 (Airsense 1996).

### 2.5.2 LDA

LDA is a traditional statistical technique for dimensionality reduction. It has been widely used and proven successful in a lot of applications. This is a classification procedure in which the classes are considered to have normal distribution and equal dispersion (covariance matrix). The goal of the analysis is to separate the classes by projecting the samples from  $p$ -dimensional space onto a finely orientated line. For a  $K$ -class problem,  $m = \min(K-1; p)$  different lines will be involved. That is to say the projection can be accomplished by  $m$  eigenvalues and  $m$  corresponding linear functions. Furthermore, LDA and PCA are all projection methods, but the LDA procedure maximizes the variance between categories and minimizes the variance within categories in order to optimize the resolution between classes. This pattern recognition analysis was

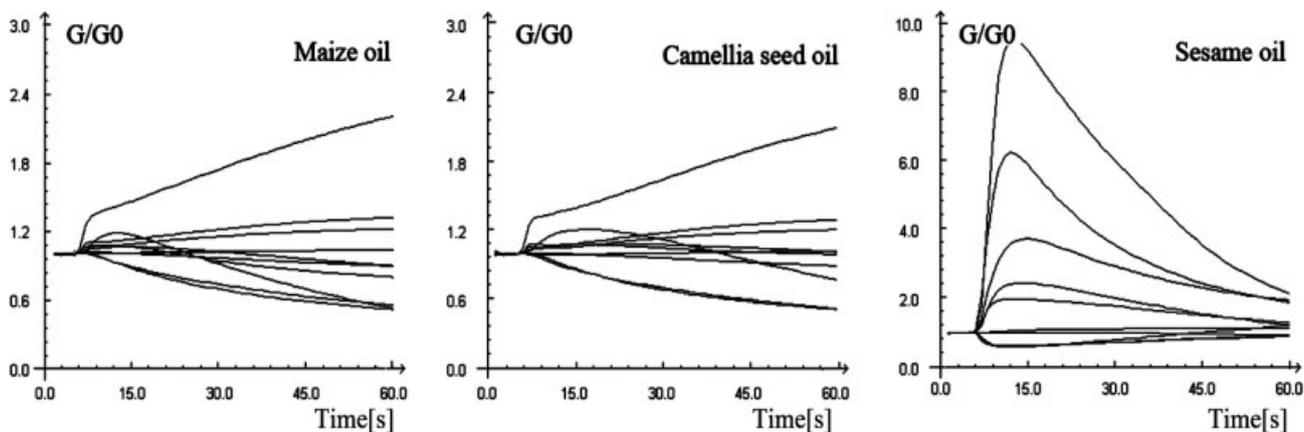


Fig. 1. Typical recordings for sesame oil, camellia seed oil and maize oil.

performed on WinMuster 1.6 (Airsense 1996). Furthermore, for calculating the Mahalanobis distance between different kinds of samples based on the linear functions, the canonical discriminant analysis was also performed on SPSS 12.0 (SPSS Inc.).

### 2.5.3 ANN

ANN can be defined as a set of very simple calculation units (nodes) that start out from a data set and transform it into a set of response values. In chemometrics, neural networks have been used to solve problems of both supervised and unsupervised pattern recognition. An ANN offers a chemometric technique of great potential for the treatment of the signals generated by electronic noses based on sensors that afford non-linear responses. For classification purposes, the network builds a model based on a set of input objects (the training set) with known outputs, adjusting the weights associated with each connection so that output values as similar as possible to the real values are generated. In order to quantitatively predict the adulteration of maize oil in sesame oil, we have employed the ANN with the Standard Back Propagation Algorithm using the neural network package in Matlab7.0 (MathWorks, Inc., 2004).

## 3 Results and discussion

### 3.1 Multivariate analysis of variance

Prior to LDA, preliminary work of multivariate analysis of variance should be performed, and only when the mean vectors of different classes are statistically different, the

LDA analysis will make sense [24]. First, Cochran's C and Bartlett's tests were applied to determine whether or not there were statistically significant differences between the standard deviations. The multiple range tests were applied to compare mean vectors when there were statistically significant differences amongst the standard deviations. The data corresponding to 15, 30 and 60 s were singled out as representatives of the signals for multivariate analysis of variance, the rest of the data were discarded.

Results of multivariate analysis of variance show that the mean vectors of different kinds of oil are statistically different from each other ( $p < 0.001$ ). Mean vectors of camellia seed oil samples with different adulteration level are statistically different ( $p < 0.001$ ), and mean vectors of sesame oil samples with different adulteration level are statistically different as well ( $p < 0.001$ ). These indicated that the sensors have different responses to different kinds of samples and maybe it is possible to differentiate the samples by their category or adulteration level. The difference of mean vectors among different kinds of oil is more prominent at 15 s than at the two other time points, 30 and 60 s, so the data of 15 s were singled out for further pattern recognition analysis.

### 3.2 Discrimination of different kinds of oil

The data of 15 s were chosen as the input of multivariate analysis, and the plots of the first two principal components and the first two linear discriminant functions are shown in Fig. 2. For PCA and LDA analysis, ellipses correspond to the 95% confidence intervals. For PCA analysis, the samples of camellia seed oil and maize oil are

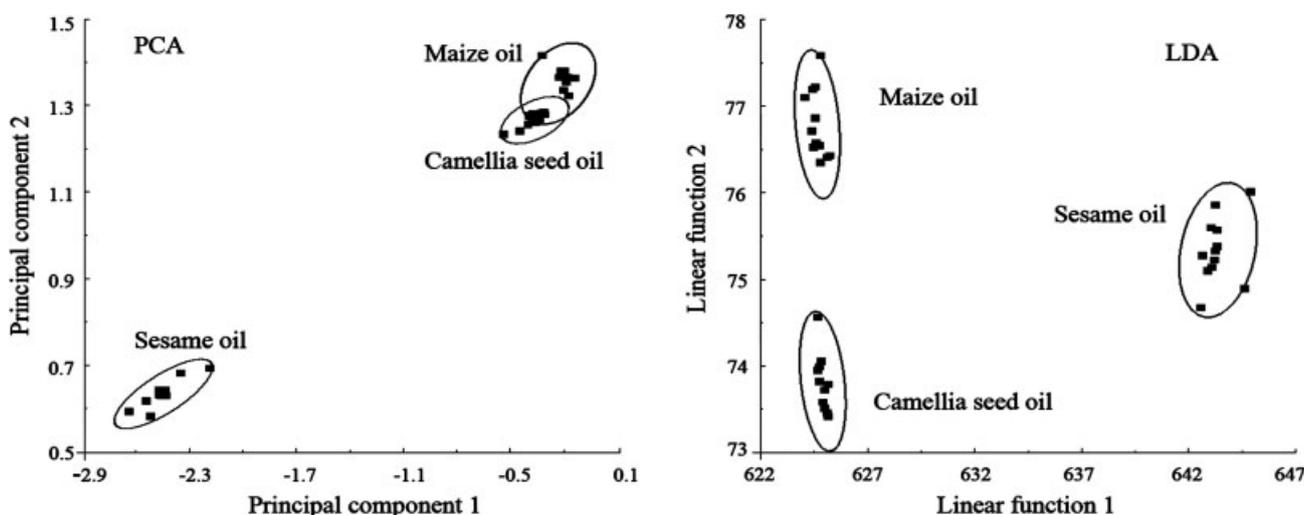


Fig. 2. Plots of PCA and LDA of sesame oil, camellia seed oil and maize oil.

more concentrative than sesame oil, but the ellipses corresponding to camellia seed oil and maize oil overlapped a little with each other, and the sesame oil can be clearly discriminated from the camellia seed oil and the maize oil. This indicates that the sensors' responses to camellia seed oil and maize oil are much alike with each other and have a big diversity from those of sesame oil. Since the LDA procedure maximizes the variance between categories and minimizes the variance within categories in order to optimize the resolution between classes, it was supposed that better results could be obtained. For LDA analysis, all of the three kinds of samples can be distinguished completely. This may be the result of the maximization of variance between categories and the minimization of variance within categories by LDA.

### 3.3 Detection of adulteration in camellia seed oil

The score plots of PCA and LDA of samples with different adulteration levels were shown in Fig. 3. In the plot of PCA, ellipses corresponding to samples with different adulteration levels overlapped with each other and this presented difficulties in discrimination. The result of LDA was much better. It was clear that, in the plot of LDA, camellia seed oil and maize oil can be separated completely from their mixtures. The samples at 10, 40 (or 50), and 80% adulterated with maize oil can be clearly discriminated from other samples or each other. The ellipses corresponding to samples with levels of adulteration of 60% and 40, 50, 80% somewhat overlapped. The ellipses corresponding to the samples with adulteration of 50 and 40% overlapped a lot and it was difficult to discriminate the samples from each other, but they can be clearly dis-

criminated from the samples with 80% adulteration. Unfortunately, if some samples with different adulteration levels are included in the pattern, there will be more complexity in the plot and more difficulty in discrimination.

In order to check the discriminative power of LDA, canonical discriminant analysis was also performed on SPSS 12.0 (SPSS Inc.). In this procedure, for the detection of adulteration in camellia seed oil, 165 samples (15 samples of maize oil, 15 samples of pure camellia seed oil and 135 samples of camellia seed oil with different adulteration levels) were separated into two subsets: 110 samples for training and the remaining 55 samples for testing. Of each group, 10 samples were included in the training set, and 5 samples of each group were included in the test set. The discriminant functions were derived from the cases of the training set; the Mahalanobis distance was calculated based on linear functions for classification. The eigenvalues and corresponding percentages of variance are shown in Tab. 2 and linear function coefficients are shown in Tab. 3. Acceptable results were obtained: Five samples in the training set and nine samples in the test set were misclassified and the accuracy was 83.6%.

### 3.4 Detection of adulteration in sesame oil

The result of PCA was shown in Fig. 4. The ellipses allowed a clear separation of the adulterated and non-adulterated sesame oils. Although the ellipses overlapped in some cases, the samples of different adulteration levels could be generally discriminated from each other, especially when the diversity between the two adulteration levels is great

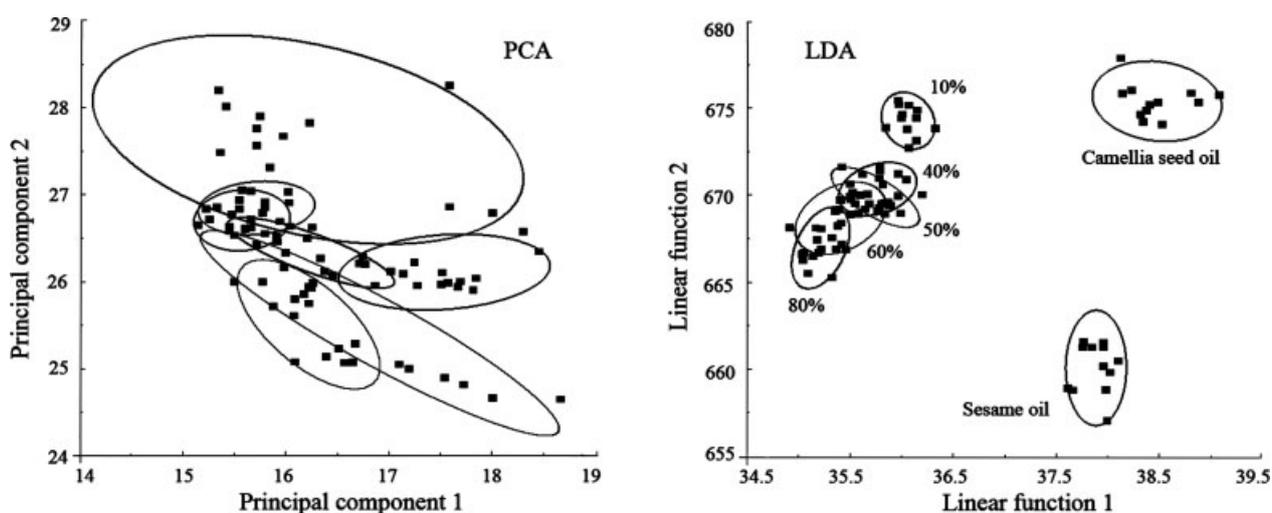


Fig. 3. Plots of PCA and LDA of camellia seed oil, maize oil and their mixtures.

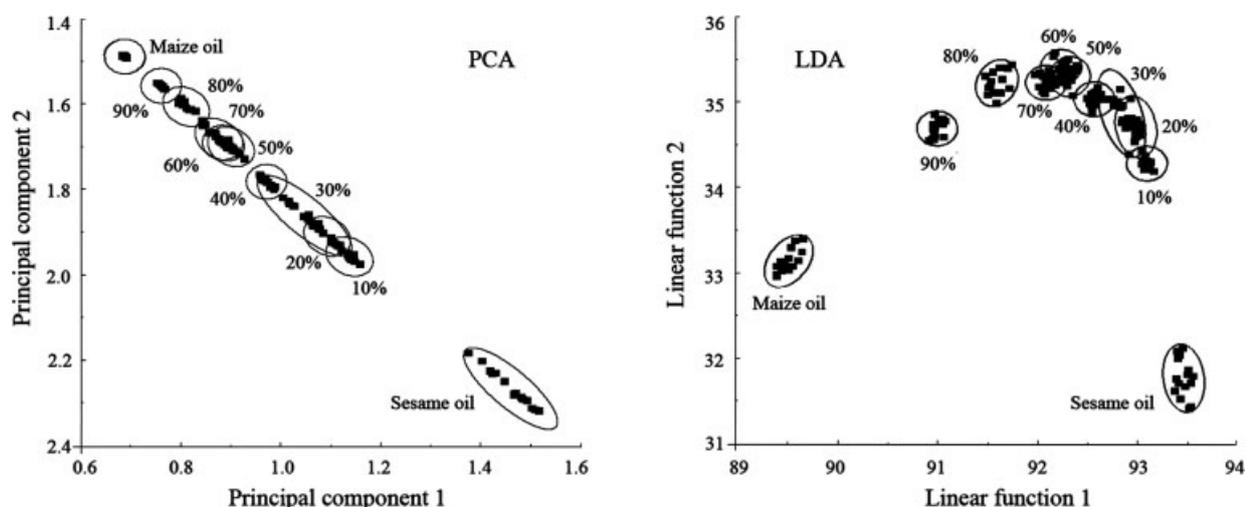


Fig. 4. Plots of PCA and LDA of sesame oil, maize oil and their mixtures.

Tab. 2. Eigenvalues and corresponding percentages of variance in the procedure of canonical discriminant analysis for detecting adulteration in camellia seed oil.

Function	Eigenvalue	% of Variance	Cumulative %	Canonical correlation
1	85.448	64.407	64.407	0.994
2	23.249	17.524	81.932	0.979
3	10.054	7.579	89.51	0.954
4	6.391	4.818	94.328	0.93
5	5.351	4.034	98.362	0.918
6	1.366	1.03	99.392	0.76
7	0.544	0.41	99.801	0.594
8	0.137	0.103	99.904	0.347
9	0.122	0.092	99.996	0.329
10	0.005	0.004	100	0.072

enough. For example, the ellipses corresponding to 90, 70, 40 and 10% adulteration were well separated from each other. The analysis data of 70, 60 and 50% adulteration overlapped with each other completely and discrimination was difficult. Adulteration levels of 30 and 20% overlapped with each other and could also not be discriminated from each other.

For the LDA analysis in Fig. 4, maize oil and sesame oil were well separated from the mixtures. Although the ellipses overlapped in some cases (60 and 50%, 30 and 20% could not be discriminated very well), the other samples of different adulteration levels could be clearly discriminated from each other. Generally, the result of LDA is a little better than that of PCA.

Tab. 3. Canonical discriminant function coefficients for detecting the adulteration in camellia seed oil.

	Function									
	1	2	3	4	5	6	7	8	9	10
Sensor 1	72.36	203.1	51.12	-62.44	-6.12	4.44	28.6	54.6	-25.86	59.64
Sensor 2	28.15	-8.37	-1.71	21.95	38.71	9.64	-3.24	38.48	-25.45	13.22
Sensor 3	-114.65	-309	-88.82	315.43	142.96	45.85	-468.91	-150.75	149.84	-543.58
Sensor 4	84.76	0.15	2.43	-53.09	8.78	18.02	27.15	-7.01	-10.65	8.85
Sensor 5	-73.47	-18.33	108.07	-215.02	-85.13	-88.82	579.86	78.78	-160.16	513.94
Sensor 6	-98.29	43.85	122.86	-125.7	4.08	42.82	3.95	56.16	0.94	37.25
Sensor 7	-15.61	17.92	30.8	80.31	-65.14	32.39	32.55	22.39	118.42	-83
Sensor 8	-59.98	57.82	-56	83.98	-20.48	29.7	71.3	-54.61	-39.26	-28.89
Sensor 9	-79.34	26.28	-43.68	-116.26	11.95	-120.53	13.09	-114.76	23.91	-17.29
Sensor 10	139.46	-73.8	37.46	66.96	40.2	22.33	-58.65	46.89	48.37	70.66

Similar to the analysis for adulteration in camellia seed oil, the canonical discriminant analysis was also performed to detect adulteration in sesame oil. The eigenvalues and corresponding percentages of variance were shown in Tab. 4 and linear function coefficients are shown in Tab. 5. The result obtained was: Two samples in the training set and three samples in the test set were misclassified and the accuracy was 94.5%.

### 3.5 Prediction of ANN

In order to predict the percentage of adulteration in camellia seed oil, a BP-net was established whose properties were shown in Tab. 6. With a similar procedure as in the canonical discriminant analysis, the BP-net was trained by 110 samples of the training set and validated by 55 samples of the test set.

The data were imported into the BP-net whose structure is 10–10–1, and after training and simulating, the results of simulation obtained were shown in the Fig. 5a. In this

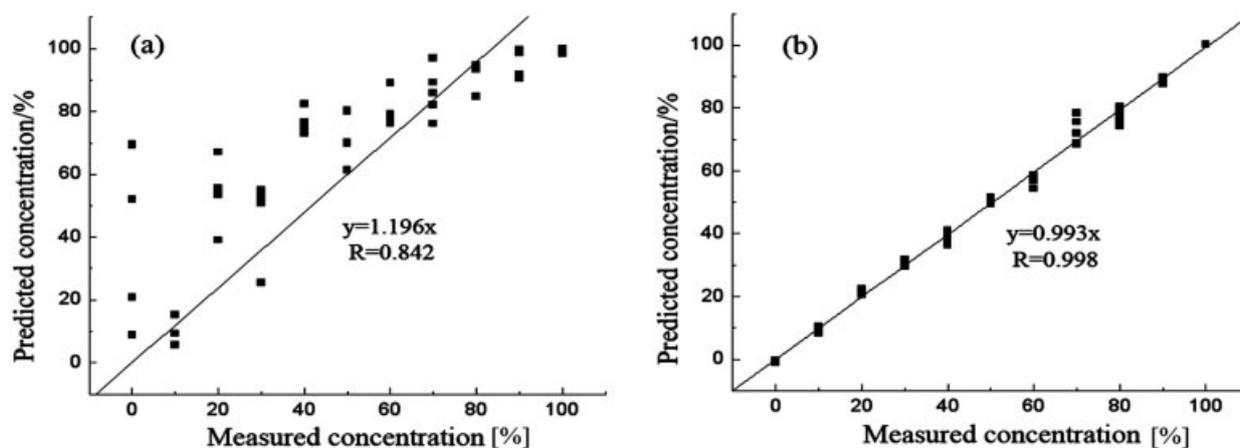
case, poor results have been obtained by ANN. Except for some special cases, the prediction of percentage is inaccurate. Furthermore, several BP-nets with different structure have been used, but none of them prevailed to

**Tab. 4.** Eigenvalues and corresponding percentages of variance in the procedure of canonical discriminant analysis for detecting adulteration in sesame oil.

Function	Eigenvalue	% of Variance	Cumulative %	Canonical Correlation
1	471.272	81.11	81.11	0.999
2	73.133	12.587	93.697	0.993
3	29.049	5	98.696	0.983
4	5.097	0.877	99.574	0.914
5	1.769	0.305	99.878	0.799
6	0.541	0.093	99.971	0.592
7	0.139	0.024	99.995	0.349
8	0.023	0.004	99.999	0.149
9	0.004	0.001	100	0.067
10	0.001	0	100	0.029

**Tab. 5.** Canonical discriminant function coefficients for detecting the adulteration in sesame oil.

	Function									
	1	2	3	4	5	6	7	8	9	10
Sensor 1	-302.72	203.91	209.08	-36.96	-32.04	43.85	18.15	-11.11	-1.06	14.88
Sensor 2	-3.44	0.71	0.88	-4.75	4.5	-2.65	-2.84	-4.39	-0.85	1.3
Sensor 3	1021.03	-918.19	-844.64	639.2	-632.12	-226.39	-394.17	-331.5	-151.33	15.74
Sensor 4	-76.13	-42.95	38.03	108.82	-78.36	-0.64	324.99	-76.77	149.81	26.87
Sensor 5	-576.74	763.47	713.49	-512.08	828.64	206.84	414.44	420.96	188.26	-15.4
Sensor 6	-5.35	17.51	-5.05	43.06	-58.72	21.72	-10.01	9.2	0.62	45.49
Sensor 7	13.92	2.72	-0.14	8.68	-10.11	0.88	13.77	2.81	-14.22	-8.13
Sensor 8	2.66	20.63	-52.72	-28.08	62.89	-20.93	-2.7	7.2	4.89	-32.24
Sensor 9	19.93	0.84	25	16.17	-3.56	8.6	3.48	8.65	8.53	0.49
Sensor 10	20.12	1.06	57.73	-32.51	15.5	129.11	-113.49	-26.5	-80.83	-11.64



**Fig. 5.** Calibration curves for samples of camellia seed oil (a) and sesame oil (b) adulterated with maize oil.

**Tab. 6.** Primary properties of the BP-net.

Net.trainparam.epochs	1000
Net.trainparam.goal	0.01
Net.trainparam.time	Inf
Net.trainparam.min_grad	1.00E-06
Transfer function of layer 1	TANSIG
Transfer function of layer 2	PURELIN
Training function	TRAINLM
Performance function	MSE
Adaption learning function	LEARNGDM

improve the results to an acceptable extent. It is impossible to predict the camellia seed oil adulteration level by using these electronic nose signals and a BP-net as the pattern recognition technique.

When the same experiments (included: preparation of samples and calculation of ANN) were conducted with sesame oil, the prediction of adulteration by ANN was a completely different case. It was possible to predict the sesame seed oil adulteration level by using these electronic nose signals. Several BP-nets with different structures have been employed, and the best result came from the net whose structure was 10–14–1. Perfect results were obtained and can be seen in the Fig. 5b. By means of SAS8.0, the 95% individual confidence interval of absolute error (–3.4416%, 5.5411%) was obtained. Based on ANN as its pattern recognition technique, the electronic nose can quantitatively predict, at least approximately, the maize oil adulteration in sesame oil.

## 4 Conclusions

An electronic nose based on ten metal oxide semiconductor sensors has been used to discriminate sesame oil, maize oil and camellia seed oil and to detect adulteration in sesame oil and camellia seed oil. Results of multivariate analysis of variance showed that the signals corresponding to different samples are significantly different.

All three kinds of oils can be discriminated by LDA, but it is difficult to discriminate camellia seed oil from maize oil by PCA. It is impossible to detect maize oil adulteration in camellia seed oil by PCA. Although better results have been obtained by LDA, some kinds of samples could not be clearly discriminated from each other. The results of detection of adulteration in sesame oil are more acceptable, and except for particular cases, samples of different adulteration levels can generally be differentiated from each other. In the process of canonical discriminant analysis, acceptable results were obtained for adulteration of both camellia seed oil and sesame oil.

In this case, based on ANN as its pattern recognition technique, the electronic nose cannot be used to predict the percentage of adulteration in camellia oil; however, it can be used to determine, at least approximately, the amount of adulteration in sesame oil.

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