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Electronic nose technique potential monitoring mandarin maturity

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Abstract

Over the past years, electronic nose technology opened the possibility to exploit information on behavior aroma to assess fruit ripening stage. The objective in this study was to evaluate the capacity of electronic nose to monitoring the change in volatile production of mandarin during different picking-date, using a specific electronic nose device (PEN 2). Principal component analysis (PCA) and linear discriminant analysis (LDA) were used in order to investigate whether the electronic nose was able to distinguish among different picking-date (ripeness states). The loadings analysis was used to identify the sensors responsible for discrimination in the current pattern file. The results obtained prove that the electronic nose PEN 2 can discriminate successfully different picking-date on mandarin using LDA analysis. But, electronic nose was not able to detect a clear difference in volatile profile on mandarin using PCA analysis. During external validation using LDA was obtained to classified 92% of the total samples properly. Some sensors have the highest influence in the current pattern file for electronic nose PEN 2. A subset of few sensors can be chosen to explain all the variance. This result could be used in further studies to optimize the number of sensors.

Keywords: Electronic nose; Non-destructive method; Maturity; Mandarin

1. Introduction

In recent years, extensive research has been focused on the development of non-destructive techniques for measuring quality attributes of fruit. In fact the quality concept is mainly related to the consumer perception and preference for foods. The consumer perception is based on the application of the five senses and for this reason the instrument "par excellence" to determine the quality are the human senses. Actually, panels of trained people are used to fix and label the criteria of quality, to assess the quality of food, and to help in the development of new products. From an instrumental point of view there is an obvious correlation between the human senses and the application of optical, chemical and tactile sensors. For several years the instrumental measure of the fruit quality has been mostly based on the basis of rheological properties such as texture and firmness [1].

The main disadvantage of the majority of these techniques is that they are not practical for cultivars or storage stations. Moreover, most of them require the destruction of the samples used for analysis. This is why, nowadays, optimal harvest dates and predictions of storage life are mainly based on practical experience, but, let these critical decisions to subjective interpretation implies that large quantities of fruit are harvested too soon or too late and reach consumer markets in poor condition.

In particular, many researches have been focused on the development of non-destructive techniques for measuring quality attributes of fruit. Among them aroma sensing are particularly promising to provide information on those parameters affected by the overall fruit quality.

A strategy for determining the state of ripeness consists of sensing the aromatic volatiles emitted by fruit using electronic olfactory systems [2]. These systems are concerning with the exploitation of the information contained in the headspace of fruits, they have been studied in the recent past with the conventional analytical chemistry equipment, and

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the correlation between the state of over-ripening and the fruit aroma has also been found both in quantitative and qualitative terms. Beside, some specific compounds have been identified as the responsible of the aroma of particular fruit.

In the last decade, the electronic nose technology has opened the possibility to exploit, from a practical point of view, the information contained in the headspace in many different application fields. Among them, food analysis is certainly one of the most often practiced.

The electronic nose offers a fast and non-destructive alternative to sense aroma, and, hence, may be advantageously used to predict the optimal harvest date. Commercially available electronic noses use an array of sensors combined with pattern recognition software. There have been several reports on electronic sensing in environmental control, medical diagnostics and the food industry [3,4]. Some authors reported positive applications of electronic nose technology to the discrimination of different fruits quality, and many experiments were performed, such as: testing orange [5], melons [2,6], blueberries [7], pears [8,9], peaches [10–12], bananas [13], apples [11,14–16] and nectarines [12].

The objectives in this research are: (1) to evaluate the capacity of electronic nose monitoring mandarin maturity during the different harvest periods, using a specific electronic nose device (PEN 2) based on sensor array and suitable pattern recognition techniques; (2) to study principal component analysis (PCA) and linear discriminant analysis (LDA) techniques to obtain whether the electronic nose be able to distinguish different ripeness; (3) to identify the sensors responsible for a discrimination in the current pattern file, using loading analysis.

2. Materials and methods

2.1. Experimental material

Chinese variety, Satsuma mandarin “Zaojin Jiaogan” (*C. reticulata*) was selected to the experiment. All the samples were hand harvested in 2003 from the experimental orchard in Department of Horticulture, Zhejiang University. Mandarin were harvested at five different picking-dates with 15 intermittent days: September 19 (the first picking-day, day 0), October 3, 18, 31 (the second, third and fourth picking-day; were expressed as day 15, day 30, day 45 and day 60, respectively) and November 15 (the five picking-day). Eighty mandarin fruits each group, and a total of 400 nose measurements were performed.

During external validation, the same variety, Satsuma mandarin, was selected to the experiment. But the samples were hand harvested from other orchard (Jingde orchard, Zhejiang), 12 km far from the experimental orchard in Department of Horticulture. Mandarins were harvested at the five same picking-dates with 15 intermittent days: September 19, October 3, 18, 31 and November 15 in 2003. Twenty mandarin fruits each group, and a total of 100 nose measurements were performed for external validation.

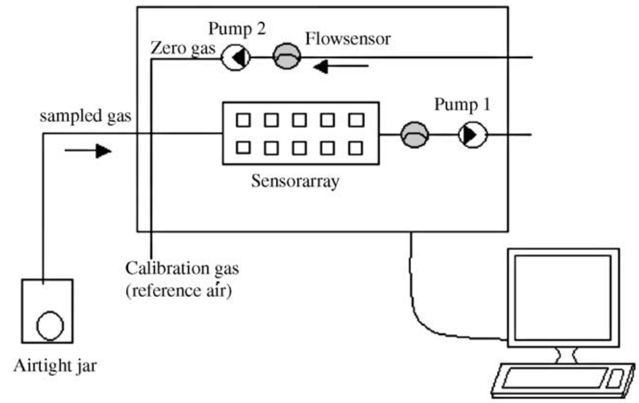

Fig. 1. Schematic diagram of the electronic-nose measurements and gas flow of PEN 2 during the experiments.

Because fruits were harvested randomly from different trees, pooled, then the experimental design was completely randomized with each fruit as an experimental unit. All fruits of each sample were individually numbered.

2.2. Electronic nose data acquisition and analysis

An electronic nose device PEN2, provided by (WMA Airsense Analysentechnik GmbH) Schwerin, Germany, was used. The portable electronic nose PEN2 has an array of 10 different metal oxide sensors positioned into small chamber ($V = 1.8$ mL). In Fig. 1 shows schematic diagram of the electronic-nose measurements and gas flow of PEN 2 during the experiments. Table 1 lists all used sensors and their main applications. This table contains current known or specified reaction.

Each fruit was placed into an airtight glass jar with a volume of 1 L (concentration chamber). The glass jar was then closed and the headspace inside it was equilibrated for 1 h. Preliminary experiments showed that after 0.5 h of equilibration the headspace reached a steady state and experiments were conducted after 0.5 h of equilibration. One luer-lock needle (20 g) connected to a Teflon-tubing (3 mm) was used to perforate the seal (plastic) of the vial and to absorb the air accumulated inside it, during the measurements. The headspace gas was pumped over the sensors of the electronic-nose with a flow of 400 mL/min; during the measurements process, three different phases can be distinguished: concentration, measurement and stand-by. The electro-valves, controlled by a computer program, guide the air though different circuits depending on the measurement phase. No matter the phase, airflow is always kept constant though the measurement chamber. During the measurement phase, the bomb pushes the volatiles though a closed loop that includes the measurement and concentration chambers. No air enters or exits the loop. The measurement phase lasts 60 s, time enough for sensors to reach a stable value. The collected data interval was 1 s.

Table 1
Sensors used and their main applications in PEN 2

Number in array	Sensor-name	General description	Reference
1	W1C	Aromatic compounds	Toluene, 10 ppm
2	W5S	Very sensitive, broad range sensitivity, react on nitrogene oxides, very sensitive with negative signal	NO_2, 1 ppm
3	W3C	Ammonia, used as sensor for aromatic compounds	Benzene, 10 ppm
4	W6S	Mainly hydrogen, selectively, (breath gases)	H_2, 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_3, 100 ppm
7	W1W	Reacts on sulfur compounds, H_2S 0.1 ppm. Otherwise sensitive to many terpenes and sulfur organic compounds, which are important for smell, limonene, pyrazine	H_2S, 1 ppm
8	W2S	Detects alcohol's, partially aromatic compounds, broad range	CO, 100 ppm
9	W2W	Aromatics compounds, sulfur organic compounds	H_2S, 1 ppm
10	W3S	Reacts on high concentrations >100 ppm, sometime very selective (methane)	CH_3, 10 CH_3, 100 ppm

When a measurement is completed, a stand-by phase is activated (60 s). The purpose is to clean the circuit and return sensors to their baseline. Clean air enters the circuit, crosses the measurement chamber first, the empty concentration chamber afterwards, and pushes the remaining volatiles out of the circuit.

Sensors were held at the temperature of 20 °C and 50–60% RH during all experiments, the temperature was maintained constant with an accuracy of ±1 °C. When the sensors are exposed to volatiles, during the measurement phase, the computer records the resistance changes that the sensors experience. When the measurement was completed, the acquired data was properly stored for later use.

The set of signals of all sensors during measurement of a sample is a pattern. Pattern of multiple measurements dealing with the same problem are stored in a Pattern File and act as the Training Set. The pattern data were recorded, checked visually and analyzed using WinMuster (version 1.5.2.4 Jun 2003, copyright 1996–2002 WMA Airsense Analysentechnik GmbH 2003).

2.3. Principal component analysis, linear discriminant analysis and loadings analysis

Pattern recognition algorithms and data processing techniques are a critical component in the implementation, development and successful commercialization of Electronic Nose (EN) systems. There are a large amount of pattern recognition techniques available. In order to select the appropriate pattern recognition algorithm for EN application, it is important to understand the fundamental nature of the data being analyzed. Statistical and non-parametric analysis techniques are the most known and commonly used to analyze EN data.

Classical statistical methods, using a probability model, were first developed and used in the field of applied mathematic, now called chemometrics. Several mathematical methods could be applied to the multi-component analysis of odors. Categorization of classifiers, can be made based on certain features, such as supervised or unsupervised, model based on model-free, and qualitative or quantitative analysis. Discriminant function analysis (DFA) is a parametric learning classifier, which can be used for both qualitative and quantitative analysis. There are many ways of performing DFA, but the classical approach is loading discriminant analysis (LDA). Principal components analysis (PCA) is a non-parametric projection method and is often used to implement a linear supervised classifier, in conjunction with discriminant analysis. This technique has been widely used for researcher to display the response of an EN to simple and complex odors and it provides qualitative information for EN pattern recognition file [17].

Using the principal component analysis (PCA) the measured data, previously trained will be transformed into 2D or 3D coordinates. This is carried out through the data reduction that extracts the most important information from the database as a result. The results of training phase can be displayed in a two dimensional view. PCA is based on a linear project of multidimensional data into different coordinates based on maximum variance and minimum correlation [18]. Training pattern from measurements of similar samples will be located close to each other after transformation. Hence, the graphical output can be used for determining the difference between groups and comparing this difference to the distribution of pattern within one group.

The linear discriminant analysis (LDA) is the first step of the discriminant function analysis (DFA). The LDA calculates the discriminant functions and similar to the PCA—a 2 or 3 dimensional display of the training set data. The difference between PCA and LDA is, that PCA does not care about the relation of a data points to the specified classes, while the LDA calculation uses the class information that was given during training. The LDA takes care about the distribution within classes and the distances between them. Therefore, the LDA is able to collect information from all sensors in order to improve the resolution of classes.

The sum of displayed variances is higher; the further principal components also contain discriminant information using PCA and LDA.

The loadings analysis is well correlated to the PCA. Using this analysis the sensors can be investigated for their responsibility for the discrimination given by the trained patterns. Sensors, located near the center of the diagram (0, 0) have a minor responsibility for the distribution of pattern in the PCA plot. They may be switched off because they may have negative influence on the pattern resolution, when particular normalizations are selected. The Loadings analysis will help to identify the sensors responsible for discrimination in the current pattern file. Single sensors may be switched off for analysis as long as they have no positive influence on the identification process.

3. Results

3.1. Electronic nose response to fruit aroma

Fig. 2 shows a typical response of ten sensors during measuring mandarin fruit. Each curve represents a different sensor transient. The curves represent sensor conductivity of one sensor of array against time due to electro-valve action when the volatiles from the fruit reach the measurement chamber. In that transition, the clean airflow that reaches the measurement chamber is substituted by airflow that comes from the concentration chamber, closing a loop circuit between both chambers. It can be seen that, after an initial period of low and stable conductivity (when only clean air is crossing the measurement chamber), conductivity increases sharply and then stabilizes after 30 s. The each sensor signal generally stabilizes and was considered to use in analysis of electronic nose. In this research, the signal of each sensor at response 42 s was used in analysis of electronic nose. Fig. 3 shows the response value of each sensor in Cartesian coordinate for an example at 42 s.

3.2. Signal analysis

Fig. 4 shows the evolution of the signals generated by the sensor array. Each line represents the average signal variation of 80 mandarins respectively for one sensor of the array (10 sensors), linking to the measurements of conductance increase or decrease as picking-date that vapors from the fruit reached the measurement chamber.

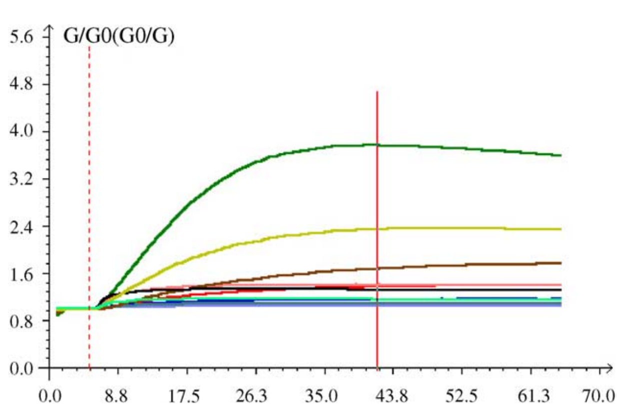

Fig. 2. Ten sensors responses to mandarin fruit aroma.

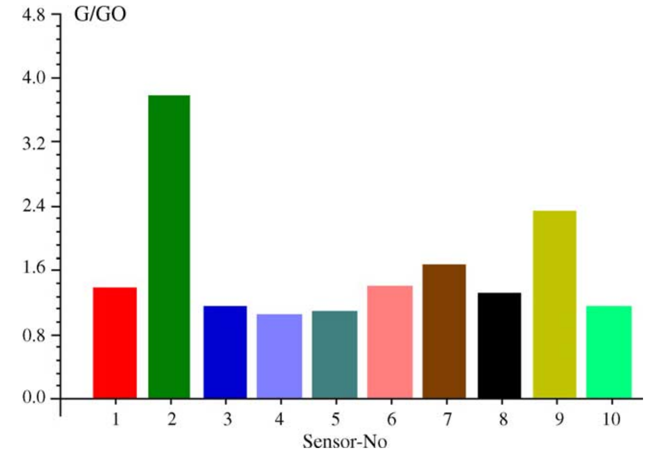

Fig. 3. Relative conductivity (G_o/G) vs. sensor number at 42 s.

With except of mandarin of picking-date (unripe mandarin), during the mandarin fruit ripeness process on the tree, the respiration decreased, meaning a decrease of the vapors generated, which vapors reach in a less quantity, the average signal of sensor array decrease (Fig. 4). The result does not agree with those obtained by Brezmes et al. testing peaches and pears [11].

It can be inferred that the sensors 2, 7, 9 have higher values, which may implied that those are important on the current pattern file and evaluated the picking-date (this is presented in paragraph 3.5 and Fig. 8).

3.3. Classification of mandarin using PCA and LDA

In order to investigate whether the electronic nose was able to distinguish among different picking-date, PCA and LDA analysis were applied in this research. The analysis was carried out using the signal stability at 42 s in mandarin.

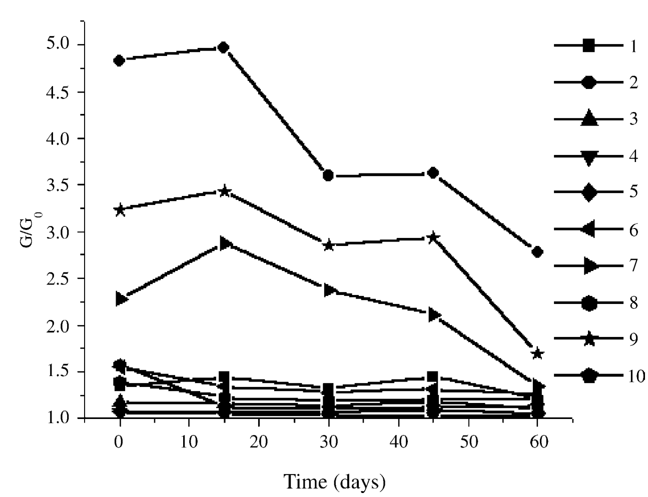

Fig. 4. Relative conductivity of each sensor vs. picking-date.

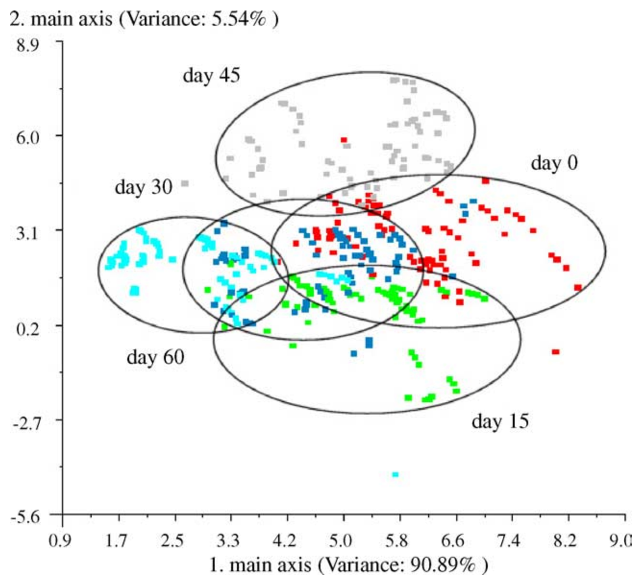

Fig. 5. PCA analysis for mandarins (80 samples).

PCA and LDA analysis results are shown in Figs. 5 and 6. Two figures show that analysis results on a two-dimensional plane, principal component 1 (PC1) and principal component 2 (PC2) in Fig. 5 and first and second linear discriminant LD1 and LD2 in Fig. 6.

PCA is a linear combinatorial method, which reduces the complexity of the data-set. The inherent structure of the data-set is preserved while its resulting variance is maximized. PCA has been performed to describe the aroma changes during picking process. Fig. 5 shows that the score plot inside the ellipses and represent the variation around each picking data (maturity state) in the space. The processed data shows a shift erratic of the different picking date along the first principal component, PC1, which explains 90.89% of the total variance with value 96.427%. The second principal component (PC2) explains 5.54% of the variation and shows no particular trend with picking date. In spite of the clear separation that was achieved among some groups (day 0, day 45 and 60) using the analysis (PCA), other groups of samples do overlap each other. The system has not enough resolution to fallow picking date or mandarin ripeness process.

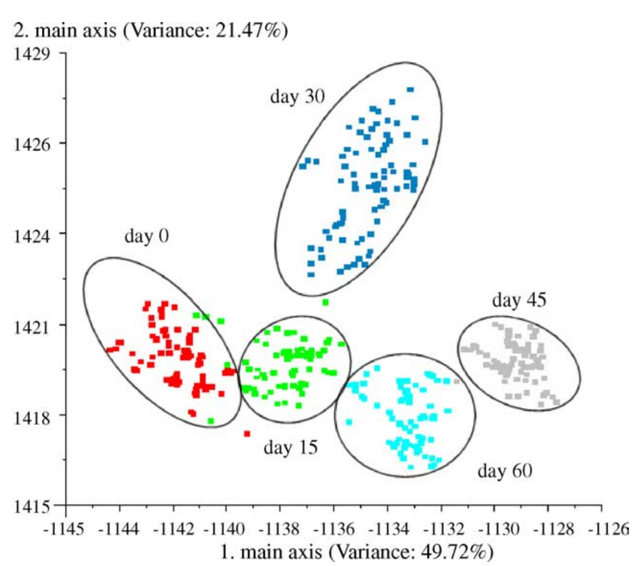

Fig. 6. LDA analysis for mandarins (80 samples).

When the LDA analysis (Fig. 6), using the same data of five groups (picking date), the fruits were clearly distinguishable from each group. In this plot about 71.193% of the total variance of the data is displayed. LDA function 1 (LD1) and function 2 (LD2) accounted for 49.725 and 21.469% of the variance respectively.

Fig. 6 shows that the classification for day 0 had one not-classified sample, this result only representing 1.25% of the total samples in this group (note: not-classified, is the given name to those samples that were located out of the class group after establish the imaginary ellipse). The second group (day 15) had six not-classified samples, representing a total variance of 7.5% of the total. Four samples were classified into group of day 0, and one sample is very near to the border of day 0; the other one of not-classified sample is located close to third group (day 30) on opposite side. The fourth group (day 45) also had one not-classified sample, which was located inside of the fifth group (day 60). The two groups of day 30 and day 60 can be classified from the other groups. The method is very efficient to differ the mandarin maturity states; also the LDA analysis was able to classify a 98% of the total samples ($n = 400$) in each respective group (five).

In spite of the clear location among all the classes by gathering date of the mandarin using the analysis (LDA), but a small overlap joint was achieved between the first group and second (day 0 and day 15), meaning that in the first 15 days the mandarin volatiles production do not differ much. This may be reason that the classification was conducted by gathering date, not by ripeness, and ripeness of some mandarins were near between in the first 15 days and second.

The variation of each group along the abscissa (LD1) with a notable increment was shown in LDA analysis; however, the group of day 60 showed an advance in negative direction on abscissa in relation with its former. The group of day 30 has also clear confines on the axis of the ordinates (LD2) in which shows a clear upward displacement along ordinate getting away from the other groups.

3.4. External validation analysis for mandarin data using LDA

External validation analysis for the new data set (100 samples) using LDA is shown in Fig. 7. Before perform the external validation analysis, all those samples that were not-classified during the training set were excluded from the pattern recognition file.

In this plot about 70.67% of the total variance of the data is displayed. LDA function 1 (LD1) and function 2 (LD2) accounted for 48.73 and 21.94% of the variance, respectively.

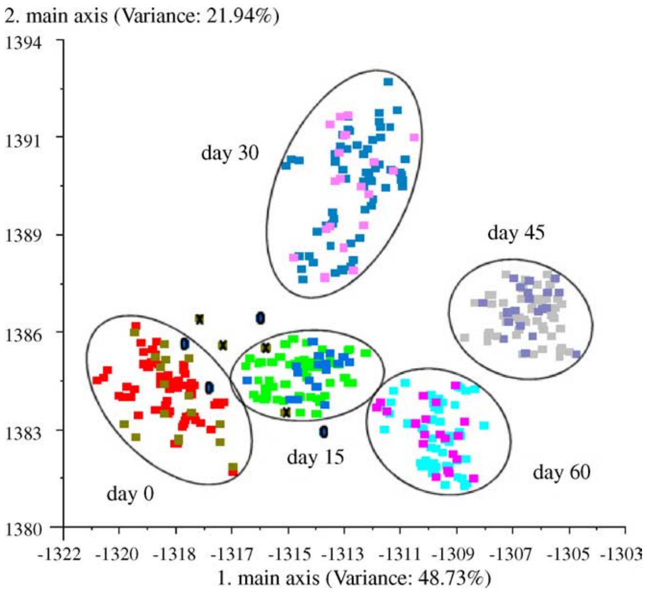

Fig. 7. LDA external validation analysis for mandarin (*x* and *o* represent the not-classified samples for day 0 and day 15 respectively).

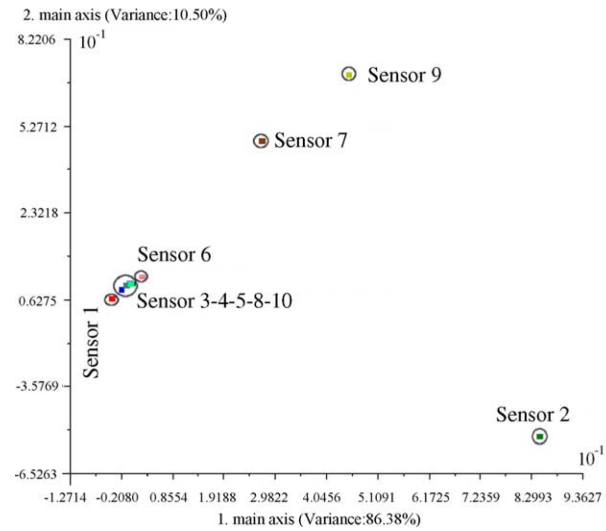

Fig. 8. Loading analysis related to PC1 and PC2 for mandarin total variance in mandarin 96.874%.

The classification for start day had four not-classified samples, both of them located into the second group (day 15); this second group (day 15) also had four not-classified samples, both of them situated into the first group (day 0). All samples corresponding to groups third, fourth and fifth (days 30, 40 and 60), were all well distributed into each respective group. Of the total of examples used for the validation set only eight of them were not-classified in their respective groups according to the different maturity states, this means only 8% of the total samples.

3.5. Loading analysis

The loading analysis will help to identify the important of sensors responsible for discrimination in the current pattern file. Single sensors may be switched off for analysis if they have rather smaller influence on the identification process. Sensors with loading parameters near to zero for a particular principal component have a low contribution to the total response of the array, whereas high values indicates a discriminating sensor.

The loading analysis was performed, a loading plot of the loading factors associate to PC1 and PC2 for mandarin shown in Fig. 8. It was also shown that the relative importance of the sensors in the array. The loading factor associates to first and second principal components for each sensor is represented. There are sensor groups that have almost identical loading parameters and they might be represented by just one sensor. Fig. 8 shows that the sensors 2, 7 and 9 have higher influence in the current pattern file, while the sensors 1, 3, 4, 5, 6, 8 and 10 have low influence. This is identical with the result in Fig. 4. The sensors 1, 3, 4, 5, 6, 8 and 10 have similar loading factor, and so could be represented by just one sensor.

Sensors with loading parameters near to dilution factor for a particular principal component have also a low contribution to the total response of the array. Hence, nearly a subset of few sensors can be chosen to explain all variance. This result could be used in further studies to optimize the number of sensors.

4. Conclusions

The obtained results prove that the electronic nose PEN 2 can differ successfully the mandarin ripeness, and have been demonstrated that electronic nose technology has excellent sensitivity and selectivity for differentiating mandarin on the basis of picking-date.

The electronic nose was not able to detect a clear difference in volatile profile on mandarin using PCA analysis; but it achieves a clear separation in all the cases using LDA analysis.

Sensors 2, 7 and 9 in mandarin have the highest influence in the current pattern file. Hence, nearly a subset of few sensors can be chosen to explain all the variance. This result could be used in further studies to optimize the number of sensors.

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Biographies

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