Evaluation of an electronic nose system for characterization of pomegranate varieties

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Abstract: The electronic nose (e-nose) would simulate the human sense (smell) to identify and realize the complex aromas by employing a chemical sensors array. One of the most common sensors used in electronic nose systems are metal oxide semiconductor (MOS) sensors. In this research, a low cost e-nose system based on six metal oxide semi-conductor (MOS) sensors as a non-destructive instrument for recognition pomegranate varieties is investigated. Principal component analysis (PCA) and linear discriminant analysis (LDA) techniques are used for this purpose. The proposed e-nose has a capability of demonstrating a clear difference in aroma fingerprint of pomegranate by PCA and LDA analysis. Using LDA analysis, it is possible to identify and to categorize the difference between pomegranate varieties, and based on the results, the classification accuracy of 95.2% was obtained. Sensor array capabilities for classification of pomegranate varieties using loading analysis were investigated too. Results showed high ability of e-nose for distinguishing between the varieties of pomegranates.

Keywords: electronic nose, linear discriminant analysis, non-destructive, pomegranate, principal component analysis

Citation: Sanaeifar, A., S. S. Mohtasebi, M. Ghasemi-Varnamkhasti, and M. M. Shafie. 2016. Evaluation of an electronic nose system for characterization of pomegranate varieties. Agricultural Engineering International: CIGR Journal, 18(3):317-323.

1 Introduction

Pomegranate (Punica granatum L.) fruit is cultivated in numerous subtropical countries particularly in the Mediterranean region. Additionally, it is cultivated widely in Iran, Afghanistan, Pakistan, India, Saudi Arabia and South America (Ekrami-Rad et al., 2011; Elyatem and Kader, 1984). The native land of pomegranate is Iran and has one of the highest area under cultivation during the time (Akbarpour et al., 2009; Khoshnam et al., 2007). Pomegranate is eaten as a fresh aril and juice, which can additionally be applied as flavoring and coloring factors. This fruit with its high antioxidant activity, provides acids, vitamins, polysaccharides, polyphenols, sugars and several essential minerals (Fadavi et al., 2005; Gil et al., 2000). Almost all elements of a pomegranate may be used. The flesh arils can be utilized as a garnish in fruit cups, compotes, salads and desserts, and also as a snack. The fruit peel is well considered for its astringent properties. The journey of fresh pomegranate between the points of harvesting and consumption includes a number of processes such as picking, sorting, packaging, storage, transportation and retailing at stores.

Technologies that classify the fruits according to their color, texture, taste, flavor and nutritive value ensure greater confidence to fruit quality, which in turn boosts the consumer acceptance and satisfaction. Most of the fruit quality measurement methods are destructive such as pulp to peel ratio determination and fruit firmness, which are mainly based on rheological properties (Ramma et al., 2001; Sanaeifar et al., 2016a). The non-destructive measurement of the fruit internal quality is becoming

Received date: 2016-05-12 **Accepted date:** 2016-08-23
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necessary for the consumers and the industry as a whole (Kheiralipour et al., 2013; Rajkumar et al., 2012).

Aroma is one of the most significant sensory attributes associated with fruits and is especially sensitive to the changes in fruit ingredients. The volatile compounds presented in pomegranate juices, can be grouped in seven chemical families: monoterpenes, aldehydes, monoterpenoids, esters, alcohols, ketones, sesquiterpenes (Melgarejo et al., 2011).

Several studies have shown the applications of e-noses including the distinction between cultivars and ripening states during shelf-life of peaches (Benedetti et al., 2008), dehydration processes of tomato slices (Pani et al., 2008), discrimination between geographical origins of orange juices (Steine et al., 2001), prediction of pears quality indices (pH, soluble solids content and firmness) (Zhang et al., 2008). Discrimination of eight different apricots varieties was considered through a number of instrumental methods. Apricots varieties were discriminated by a FOX 4000 e-nose using principal component analysis (PCA). Then aroma compounds were acquired through liquid–liquid extraction and SPME, and determined by GC–MS. Concentrations of aroma compounds were statistically studied by means of PCA and factorial discriminate analysis (FDA). PCA and FDA were able to discriminate eight varieties of apricots. A good relation between response of sensors and several fruit quality indices were observed. The outcomes demonstrate that e-noses can be a reliable tool to classify fruits (Solis-Solis et al., 2007). Research concerning pomegranate fruit using an e-nose system has not been reported. Moreover, recognition of pomegranate varieties is very difficult due to many similarities among the varieties. Thus the objective of the present research is to assess the ability of an e-nose system to distinguish three export varieties of pomegranates in Iran.

2 Materials and methods

2.1 Experimental material

The experiments were carried out with three export varieties of the best pomegranates in Iran, "Shishe Cap-e-Ferdows" or "Ferdows", "Rabab-e-Neiriz" or "Rabab" and "Malas-e-Saveh" or "Saveh" were supplied in 2012 during the fully ripened stages from field-grown trees at Khorasan, Shiraz and Markazi provinces respectively (Figure 1). The pomegranates were hand-harvested and hand-picked at a commercial orchard in order to make sure their freshness and also to prevent damage during harvesting and transporting. Before or during harvest time was not rainfall. The fruits were selected for uniformity of size and firmness, as well as freedom from any defects and mechanical damages. Samples were held in optimal conditions (5 ℃ and 85% RH) before measurement. At least 10 h before the actual measurement, the pomegranates were stored at the desired temperature.

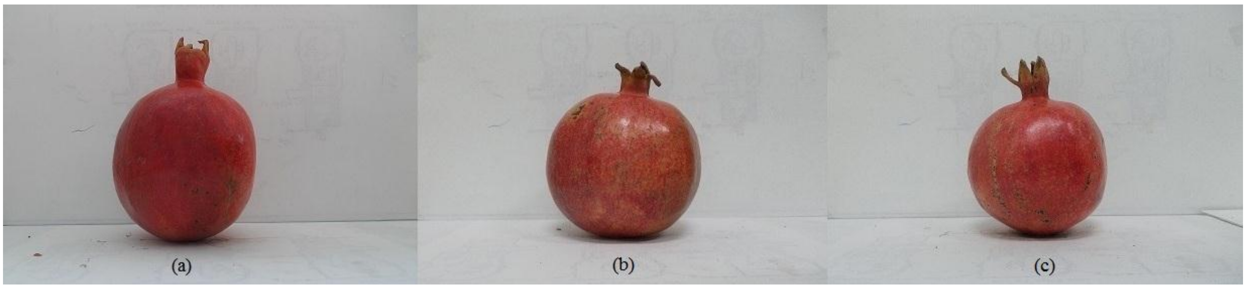

Figure 1 (a) Ferdows (b) Rabab (c) Saveh pomegranates varieties

2.2 Experimental set-up

The e-nose system consisted of a data acquisition card (NI USB-6009, National Instruments Corporation, USA) and an array of metal oxide semiconductor (MOS) sensors. A set of six gas sensors (Hanwei Electronics Co., Ltd., Henan, China) were placed in a cycloid chamber. To reach the working temperature according to the companies operating data sheets (300 ℃–500 ℃), the sensors were heated by applying a 5V DC voltage to their heater resistance. The sensor array specification utilized in the e-nose system is presented in Table 1. The experimental set-up is shown in Figure 2, and it consists of a sample chamber and a sensor chamber for the sampling system. There are different parts inside this system including one air pump, tubes, and several control valves.

Table 1 Gas sensor array of the e-nose system

Name	Main Applications	Typical Detection Ranges, ppm
MQ-3	Alcohol	0.05-10
MQ-5	LPG, Natural gas, Coal gas	200-10000
MQ-9	CO and combustible gas	20-2000 (Carbon monoxide), 500-10000 (CH_4), 500-10000 (LPG)
MQ-136	Sulfureted hydrogen	1-200
MQ-137	Ammonia	5-200
MQ-138	Organic steam	10-1000 (Benzene), 10-1000 (Alcohol), 10-3000 (NH_3)

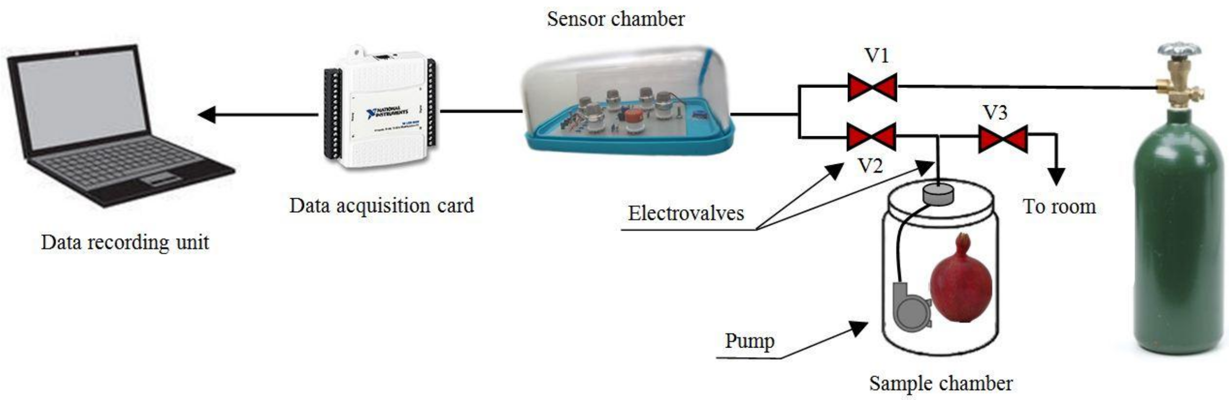

Figure 2 Experimental setup of the e-nose system

Measurement process is divided into phase's concentration, measurement, and desorption. According to the measurement phase of the system, the air is directed through the different circuits by the electro valves controlled by a computer program. All experiments were carried out at the temperature of 25 ℃ and 25%-35% RH, and the temperature was kept constant with an accuracy of ±1 ℃ during the experiments. The measurement procedure begins by locating a pomegranate inside the sample chamber. Preliminary experiments revealed that the headspace achieved a stable condition right after 1800 s of equilibration, therefore, the experiments were performed after 1800 s of equilibration and was designed to reinforce the aromatic concentration to acquire high sensor responses. As soon as the concentration phase ends, synthetic air is passed over the sensors for 200 s to reach their baseline values. Then, measurement phase was done for 120 s, which is enough for sensors to achieve a stable value. In this phase, the headspace gas was transferred to the sensors chamber utilizing a pump (the flow rate was 1.3 l/min).

The purging phase was activated after the measurement completion for 80 s. Its main purpose was to remove the odor molecules and to clean or to purge the sensors through utilization of synthetic air in such a way that the sensors could return to their baseline values and through the pump, the air existing inside the sample chamber is exhausted. Immediately after that, a new measurement is started. On the computer screen, the experimental data was displayed in real-time and saved as text files on a disk for data processing (Sanaeifar et al., 2014).

2.3 Data analysis

There are many options of pattern recognition techniques such as principal component analysis (PCA), linear discriminant analysis (LDA), cluster Analysis (CA), support vector machine (SVM), artificial neural network (ANN), fuzzy logic and etc to e-nose data analysis

(Dymerski et al., 2011). But, PCA and LDA are two well-known techniques for data classification and dimensionality reduction. They also have been widely applied and demonstrated successfully in several applications (Ouyang et al., 2013; Sanaeifar et al., 2016b). For distinguishing three pomegranate varieties using e-nose, PCA and LDA were applied. PCA is a chemometric linear, unsupervised and pattern recognition technique employed in a multivariate problem for classifying and reducing the dimensionality of numerical datasets. This method utilizes linear transformations to map data from high dimensional space to low dimensional space. The PCA effectively reduces the number of features and shows the data set in a low-dimensional subspace through removing minor components (Penza and Cassano, 2004). LDA is a supervised classification technique that maximizes the variance between categories and minimizes the variance within categories. The LDA controls the distances between classes and the distribution within them. As a result, the LDA can acquire data from all sensors in order to enhance the resolution of classes (Zhang and Wang, 2007). The difference between LDA and PCA is that PCA has most of feature classification, though LDA has data classification advantages (Balakrishnama and Ganapathiraju, 1998). The software of SPSS Statistics 21.0 (IBM, USA) and the Unscrambler 10.3 (CAMO AS, Trondheim, Norway) were employed for these analyses.

3 Results and discussion

Three pomegranate varieties were utilized to assess the proposed e-nose system. Seven different samples of each variety were collected and then the average response of seven samples was considered as the aroma fingerprint for each pomegranate variety. The resulting patterns for testing the aroma of the pomegranate samples are demonstrated in Figure 3. The value of each axis shows the fractional change in voltage $(V\text{-}V_0)/V_0$, where V_0 expresses the voltage of the sensors when the synthetic air blows over sensor array. This is an indication of the potential to employ non-specific sensor arrays to create an odor database (Tang et al., 2010).

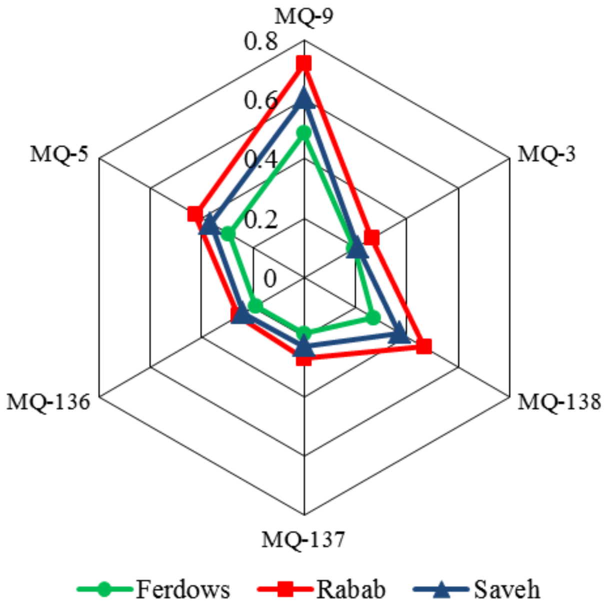

Figure 3 Aroma fingerprint varieties of pomegranate

3.1 PCA and LDA analysis

In order to evaluate the ability of the chemical sensor array to distinguish between pomegranate varieties, PCA and LDA analysis were applied to the 21 measurements performed with the e-nose, i.e. 7 measurements for each variety. PCA and LDA analysis results are shown in Figure 4. This figure represents analysis results on a two-dimensional plane, principal component 1 (PC1) and principal component 2 (PC2) in Figure 4a and the first and second linear discriminant LD1 and LD2 in Figure 4b.

By using PCA and LDA, the distinction between pomegranate varieties was well done. Rabab pomegranate variety is completely apart from the other two varieties. Groups of saveh and ferdows pomegranate varieties are somewhat closer to each other. The first two components, PC1 and PC2, contain 97% of data variance. The first principal component, PC1, explains 92% of the total variation, while 5% of the total variance is explained by PC2. The final results showed an excellent classification by LDA. In Figure 4b, about 100% of the total variance of the data is displayed and LD1 and LD2 accounted for 84.8% and 15.2% of the variance, respectively, and classification accuracies obtained by LDA method with leave-one-out cross-validation was 95.2%.

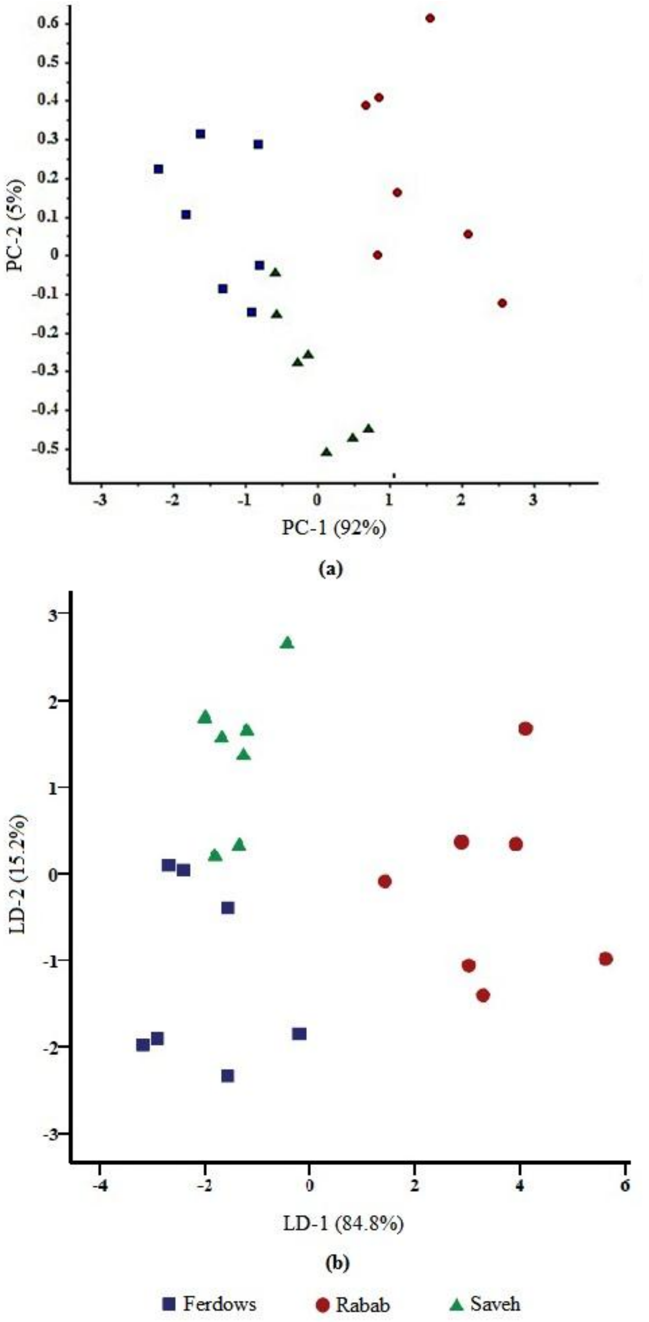

Figure 4 (a) PCA and (b) LDA results for three pomegranate varieties

3.2 Loading analysis

Loadings analysis helps to recognize the sensors liable for discrimination of pomegranate varieties in the current pattern file. Sensors with loading parameters close to zero for a particular principal component have a low contribution to the total response of the e-nose sensor array, whereas high values signify a discriminating sensor. The relative importance of the sensors in the array is displayed in Figure 5. The loading factor related to the first and the second principal components for each sensor is shown. This figure represents that the sensor array has a higher capability in the current pattern file.

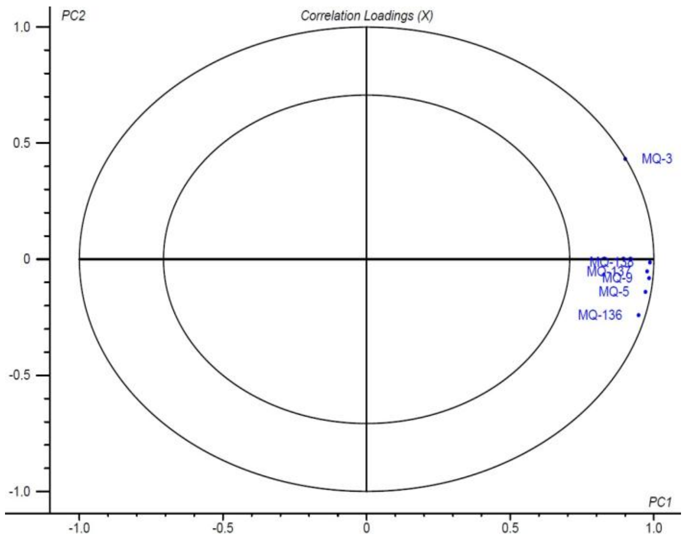

Figure 5 Loading analysis related to PC1 and PC2 for pomegranates

First class distance (Ferdows pomegranate variety) from the other two varieties is shown in Figure 6. There are great distances between three classes. Thus, three pomegranate varieties were completely discriminated from each other.

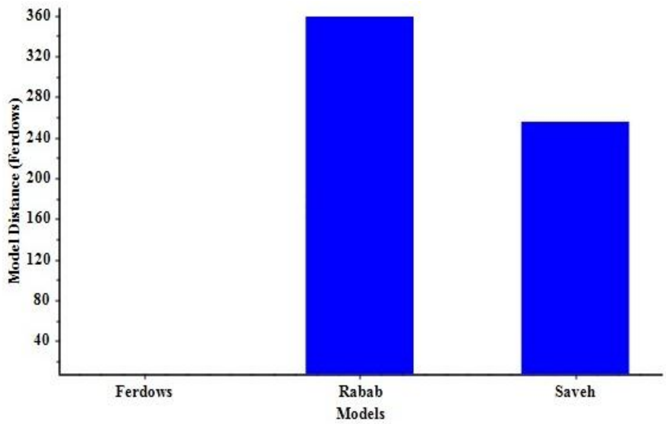

Figure 6 Ferdows pomegranate variety is completely distinct from the other pomegranate varieties

This research explores an alternative to quality control assessing in the food industry, to replace or minimize the traditional analytical methods which are high-cost, time-consuming, require the usage of environmental unfriendly chemicals, and are mainly influenced by the skills of the analyst. The e-nose system coupled with multivariate data analysis can represent an analytical tool able to provide fast information for the characterization of pomegranate directly in the packing house and retail store or in the orchard. For example, in the future, this system may provide the possibility to acquire a unique fingerprint of a given pomegranate and create a library of pomegranates based on their aroma.

4 Conclusions

In the present study, a new low cost MOS-based e-nose was evaluated. The potential of the e-nose system to characterize and distinguish the origin of three common pomegranate varieties commercialized in Iran was studied. PCA and LDA were employed to investigate whether the e-nose was able to distinguish among pomegranate varieties. By using PCA and LDA, the distinction between pomegranate varieties was conducted very well. By carrying out loading analysis, the capability of the e-nose sensors was computed, and it is concluded that the ability of the sensor array is appropriate for the aroma fingerprint recognition. Therefore, this procedure could represent a rapid, non-destructive, cheap, easy-to-use, reliable and efficient classification tool to verify the variety origin of pomegranate, not requiring chemical analyses in order to guarantee the authenticity of this product, safeguard consumers from commercial frauds and human unintentional errors in the identification. In particular, this may be a useful tool for control of protected designation of origin.

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