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Application of electronic nose with chemometrics methods to the detection of juices fraud

Mansour Rasekh | **Hamed Karami**

Department of Biosystems Engineering, University of Mohaghegh Ardabili, Ardabil, Iran

Correspondence
Mansour Rasekh, Department of Biosystems Engineering, University of Mohaghegh Ardabili, Ardabil, Iran.
Email: ma_rasekh1349@yahoo.com.au

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Abstract

Today, consumer demands for fresh foods with desirable sensory properties are increasing. Lack of easy, cheap, and nondestructive methods to control the quality of fruit juices is one of the main challenges in the beverage industry. The present study has examined two types of natural and industrial juices. Machine olfaction with 9-metal oxide semiconductor (MOS) sensors was used for the experiments. Sensor response patterns were analyzed using chemometrics methods, including principal component analysis (PCA), linear and quadratic discriminant analyses (LDA and QDA), and support vector machine (SVM). According to the results, the classification accuracy of SVM, QDA, and LDA methods was 96.25%, 95.8%, and 94.4%, respectively.

Practical applications

It is important to note that most fruit juices sold in supermarkets contain only a small percentage of natural fruit juice and usually include added sweeteners. As a result, these juices lead to the intake of large amounts of calories without providing nutrients for the body. In addition, performing chemical tests to determine the quality of fruit juices are time-consuming, destructive, and costly and requires several glassware and reagents. E-nose could be used for real-time monitoring of the volatile components of the food to evaluate different features of the product. Generally, E-nose evaluates a mixture of smells released from a sample and is a reliable, nondestructive, cost-effective, and portable method with high feasibility and speed as well as simple use.

1 | INTRODUCTION

Prevention of food adulteration has been a controversial topic in the last decade, and various intervention systems have been developed to prevent counterfeit products from entering the supply chain. Therefore, reliable analytical tools are needed to validate food products (Spink et al., 2017).

Consumers and the beverage industries have considered monitoring the quality and freshness of fruit juices. Receiving significant attention from the consumers due to the nutritional and sensory properties. Quality has become an important issue as consumers gain more awareness of all aspects related to food quality. The customers' decision to select fruit juices depends on their personal preferences (Haddi et al., 2014). Fruit juice that has lost its fiber and nutrients is actually a concentrated source of sugar that lacks most of the supplementary nutrients required to facilitate digestion and metabolism. Fruit juice increases blood sugar faster than the whole fruit, and the level of sugar obtained from fruit juice is significantly higher. It is important to note that most fruit juices sold in supermarkets contain only a small percentage of natural fruit juice and usually include added sweeteners. As a result, these juices lead to the intake of large amounts of calories without providing nutrients for the body. Preparing natural homemade juice can almost completely preserve the pulp and skin, so it is useful for human health (Jaffe & Mani, 2018).

Extensive research has recently focused on the development of non-destructive methods to assess fruit quality properties. In fact, the concept of quality is primarily related to the consumers' perceptions and preferences for foods. Consumer perception is based on the application of the five senses, which make a "superior" tool to determine the quality (Gómez et al., 2006). Olfaction is widely used by experts in industries for quality evaluation. This method is costly for the industries, whereas there are different problems, such as olfaction correct matching, time requirements, variable responses among different people, the subjective response of experts to odors, and the impossibility of applying this method to evaluate hazardous substances. The electronic nose is one of the new methods for food quality control. This system simulates the human sense of smell and uses sensor arrays to detect the effect of odor in the headspace of the samples (Karami et al., 2020).

The electronic nose is inspired by the human sense of smell and has been used as an effective tool in the quality assessment of food products. This system consists of a sensor array, a signal processing subsystem, and a pattern recognition module (Hong et al., 2015). The sensor array of the electronic nose detects odors, including large amounts of various volatile compounds in the headspace of the sample, after which the output is provided, representing the fingerprint of all sample components. The "fingerprint" described by electronic nose sensors is used to extract potential information about samples based on a suitable algorithm. The electronic nose is an automated nondestructive method, which has become a popular system due to different advantages such as convenient construction, high sensitivity, and cost-effectiveness (Qiu & Wang, 2017).

The electronic nose has been widely recommended to monitor the quality of beverages. Among different applications of the electronic nose in various research fields, considerable attention has been to quality control in the juice industries. For example, this technology has been successful in the classification and prediction of different products such as extra virgin olive oils (Teixeira et al., 2021), tangerine (Gómez et al., 2006), pineapple (Torri et al., 2010), strawberry juice (Qiu et al., 2015), apple juice (Niu et al., 2019), kiwi juice (Luo et al., 2016), mandarin juice (Qiu & Wang, 2017), orange juice (Li et al., 2020), tomato juice (Hong et al., 2015), and fruit juice spoilage (Gobbi et al., 2010) through the flavor monitoring.

Many supervised classification methods such as linear discriminant analysis (LDA), quadratic data analysis (QDA), support vector machines (SVM), and artificial neural networks (ANN) have been used successfully to analyze electronic nose data (Karami et al., 2020; Karami Rasekh, & Mirzaee-Ghaleh, 2020a, 2020b). In general, supervised classification requires labeled data to fit good classification; in other words, labeled data can represent almost the basic structure of the entire data space. However, adequately labeled data require a lot of materials, money, effort, and time. Therefore, it is important to find a classification approach with better performance using limited labeled training data (Hong et al., 2015).

Determining the quality of fruits is relatively easy through their morphological properties (such as color, texture, and firmness) and flavor (smell and taste). However, processing fruits to produce juice makes it difficult to determine the quality. For example, it is difficult to realize whether a bottle of juice is made from fresh fruits or not. Therefore, identifying and tracking the quality of natural and fresh juices will be of great importance. The present study has used an electronic nose to evaluate adulteration in natural and industrial juices. Accordingly, four different types of natural juices, including orange, lemon, mango, and strawberry from the industrial type were evaluated.

2 | MATERIALS AND METHODS

2.1 | Sample preparation

First, four types of fresh fruits, including oranges, lemons, strawberries, and mangoes, were purchased from the fruit market in Kermanshah, Iran. The samples were refrigerated until the beginning of the experiments. Then, the fruits were washed and juiced by a juice extractor. The room temperature was controlled at (20 ± 0.5)°C during sample preparation and detection.

All juices available in the Iranian market are fruit concentrate, which contains water and other additives, including citric acid, pectin, ascorbic acid, and natural flavors. This type of fruit juice is sold as natural fruit juice and contains 100% of the fruit content. Sometimes, some producers may market this juice as fresh and pure juice. Therefore, 15 pure and 15 industrial samples were prepared for each type of fruit juice, resulting in 120 samples (15 replications $\times$ 8 fruit juice groups). Twenty milliliters of each sample was placed in a 50-ml glass jar at room temperature (23 ± 2)°C for the experiment. The samples were placed in closed containers for 10 min to reach the equilibrium state of the headspace.

2.2 | Electronic nose system

Experiments were performed with an olfactory system made in the Biosystems Engineering Department of the University of Mohaghegh Ardabili. Electronic nose system includes air filter, sample chamber, solenoid valves, pump, sensor array, data collection system, and laptop, schematically presented in Figure 1 (Karami et al., 2020).

Data were collected from fruit juice samples in three phases of cleaning, measuring, and cleaning the sensors, which took a total time of 300 s. In the first phase, the air was pumped into the sensor chamber for 100 s so that the response of the sensors reached the baseline, and the system was ready for experiments. During the measurement process, the gaseous compounds of the sample headspace were pumped through a Teflon tube into the sensor array, after passing through the solenoid valves, changing the output voltage ratio of the sensors. The measurement phase lasted 100 s, which was long enough for the sensors to reach stable signal values. As shown in Table 1, the designed electronic nose system used a combination of 9 metal oxide semiconductor sensors with MQ and TGS trademarks.

The computer stored the output signal data of the sensors during the measurement period, as one data per second. After the measurement process was complete, the obtained data were stored for

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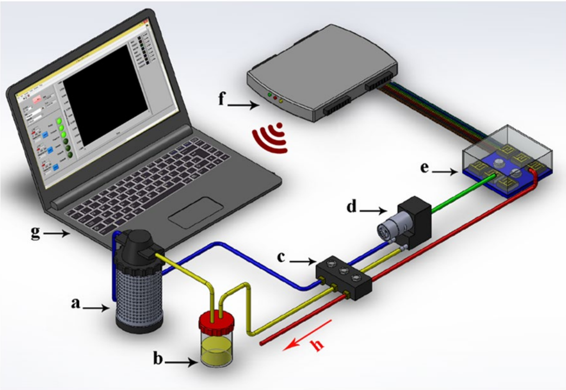

FIGURE 1 Schematic of olfactory system used (a) air filter (carbone active), (b) sample compartment, (c) solenoid valve, (d) diaphragm pump, (e) sensor array, (f) date acquisition card, (g) PC, and (h), air outlet

TABLE 1 The used sensors in electronic nose

Row	Sensor name	Detection ranges (ppm)	Main applications (gas detector)
1	MQ-9	10–1,000 and 100–10,000	CO and combustible gas
2	MQ4	300–100	Urban gases and methane
3	MQ135	10–10,000	Steam ammonia, benzene, sulfide
4	MQ8	100–1,000	Hydrogen
5	TGS2620	50–5,000	Alcohol, steam organic solvents
6	MQ-136	1–200	Sulfur dioxide (SO_2)
7	TGS813	500–10,000	CH4, C3H8, C4H10
8	TGS822	50–5,000	Steam organic solvents
9	MQ3	10–300	Alcohol

further analysis. Application of the fraction method aimed to correct the baseline. In this method, the response of the sensors becomes dimensionless in addition to normalization to eliminate noise or possible deviation (Karami, Rasekh, & Mirzaee – Ghaleh, 2020; Karami et al., 2020; Karami Rasekh et al., 2020a):

$$Y_s(t) = \frac{X_s(t) - X_s(0)}{X_s(0)}, \tag{1}$$

in which, $Y_s(t)$, $X_s(0)$, and $X_s(t)$ indicate the normalized sensor response, the baseline, and the sensor response, respectively.

After each measurement, the filtered air was pumped into the sensor chamber by activated carbon for 100 s (cleaning time) from the other port of the device. This phase aimed to discharge the odor of the chamber, bring the sensor response to the baseline, and prepare the system for further experiments. All experiments were performed by the e-nose at ambient temperature (23 ± 2)°C, with air conditioning control.

2.3 | Chemometrics analysis

The preprocessed data were analyzed using various chemometric analyses. Principal component analysis (PCA) was used to identify patterns and visualize existing information in the data measured by the electronic nose. This method can reduce the multidimensional data set to smaller dimensions without losing useful information for analysis. PCA is an orthogonal linear transformation that transforms data into a new coordinate system. Each principal component (PC) is a linear combination of the main measured variables, which are noncorrelated and arranged in a way that the first few cases retain most of the variations in all the main variables. Linear discriminant analysis (LDA) is commonly used in pattern recognition, statistics, and machine learning as a dimensional method to find a linear combination of new variables from the original data. The LDA seeks to minimize intraclass and maximize interclass variances. The support vector machine (SVM), commonly used for sample classification and regression, is a supervised learning model based on the concept of the decision planes. The root mean square error (RMSE) and correlation coefficients (R^2) are two statistical parameters used to evaluate the performance of regression models, where the higher the R^2 and the lower the RMSE, the better will be the regression model. The model inputs included data obtained from the response of nine sensors and the output included the type of fruit juice. The confusion matrix was used to select the best model. Sensitivity, specificity, accuracy, and precision parameters were used to analyze the system performance (Basri et al., 2017):

$$\text{Sensitivity} = \frac{TP}{TP + FN}, \tag{2}$$

$$\text{Specificity} = \frac{TN}{TN + FP}, \tag{3}$$

$$\text{Precision} = \frac{TP}{TP + FP}, \tag{4}$$

$$\text{Accuracy} = \frac{TP + TN}{TP + TN + FN + FP}, \tag{5}$$

$$\text{AUC} = \frac{\text{Sensitivity} + \text{Precision}}{2}. \tag{6}$$

The weight and importance of each criterion were considered equal to one. Unscrambler X 10.4 and Matlab 2014a software were also used for data analysis.

3 | RESULTS AND DISCUSSION

3.1 | PCA results

Only two features extracted from the response curve of each sensor were used to perform PCA analysis. Figure 2 shows the results of the principal component analysis and is constrained by the two principal

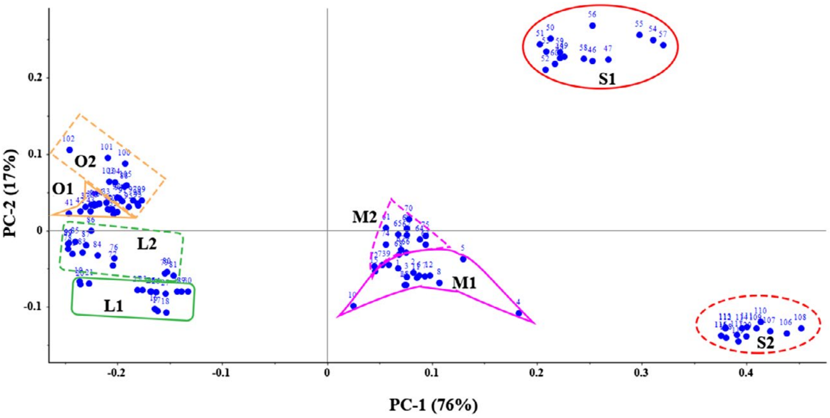

FIGURE 2 Two-dimensional PCA plot performed on eight fruit juices with data collected using electronic nose

components. According to Figure 2, it is possible to detect different juices (natural (M1) and industrial (M2) mango, natural (L1) and industrial (L2) lemon, natural (O1) and industrial (O2) orange, and natural (S1) and industrial (S2) strawberries) successfully based on the electronic nose signal responses. The scores plot is especially useful for PC1 and PC2 as they summarize more variations in the data than any other pair of PCs. Accordingly, the total variance of the data was 76% for PC-1 and 17% for PC-2, and the first two principal components constituted 93% of the total variance of the normalized data. The variance of the total PC1 and PC2 share was above 90%, indicating that the first two PCs could explain the total variance of the dataset. According to the figure, except for natural (S1) and industrial (S2) strawberries, which have a significant distance, other natural and industrial samples show a negligible distance. Thus, it can be assumed that the electronic nose has detected the smell of fruit juice instead of distinguishing different types of juice from each other. Therefore, the odors of citrus fruits (orange and lemon) are very similar to each other, whereas the odor of mango and strawberry is completely different, indicating the high accuracy of the electronic nose in detecting the smell of different products. These findings are consistent with the results obtained by other researchers (Haddi et al., 2014). Jiang et al. (2021) for qualitative identification of the edible oil storage period using PCA, they achieved 99% accuracy.

The relationships between all variables can be easily interpreted in the correlation loadings plot. The loading diagram shows the relative role of the sensors used in the electronic nose for each principal component. The encircle shows 50% and the encircle shows 100% of the variance of the data. The greater the amount of sensor loading on a principal component, the greater will be the role of that sensor in detection and classification. Therefore, that sensors placed on the encircle have a greater role in data classification. As shown in Figure 3, the highest response was related to MQ135, TGS813 sensors, whereas the lowest response was related to MQ3-4-8-9 and 136 sensors. Accordingly, the addition of sensors with good differentiation features increases the classification abilities.

3.2 | LDA and QDA analysis

Linear and quadratic (LDA and QDA) analyses were used to classify the differentiation in the fruit juice samples based on the data measured by

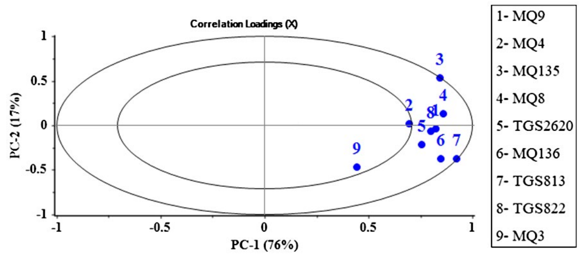

FIGURE 3 Loading plot for PCA analysis for fruit juices

the electronic nose. Compared to the principal component analysis, the LDA method can extract information from multiple sensors to optimize interclass resolution. Therefore, this method was used to classify the two groups of natural and industrial juices and eight groups of juices based on the electronic nose signals in another analysis. The results of classification based on the pure and industrial juices in the LDA and QDA methods were equal to 85.83% and 90.83%, respectively (Figure 4). Table 2 presents the confusion matrix obtained from fruit juice classification by the LDA and QDA methods. According to Equations (2) to (6), performance parameters of the LDA and QDA methods in the classification of fruit juice types can be summarized in Table 3. The confusion matrix is used to calculate the performance parameters of the detection models. According to the results of Table 3, the accuracy of data classification was 0.861 and 0.908 using the LDA and QDA methods, respectively.

The LDA and QDA analyses were also used to classify all types of juices, including eight groups. According to Figure 5, both methods showed a total detection of 92.5% and 99.17%, respectively. These results were much higher than those obtained for the 2-group classification. The confusion matrix and performance parameters of the LDA and QDA methods are presented in Tables 4 and 5 for the classification of all types of juice. According to the results of Table 4, the accuracy of data classification was 0.928 and 0.992 for the LDA and QDA methods, respectively. Therefore, the LDA and QDA methods performed intragroup classification well and with high accuracy. In general, according to the results, the QDA method has been more accurate than the LDA method in data classification. In a study on the shelf life of edible oil, the accuracy of data classification was 94.4% and 95.8% for the QDA and LDA methods, respectively (Gómez et al., 2006). In another study, the LDA and QDA methods were used to classify apples based on their storage time using

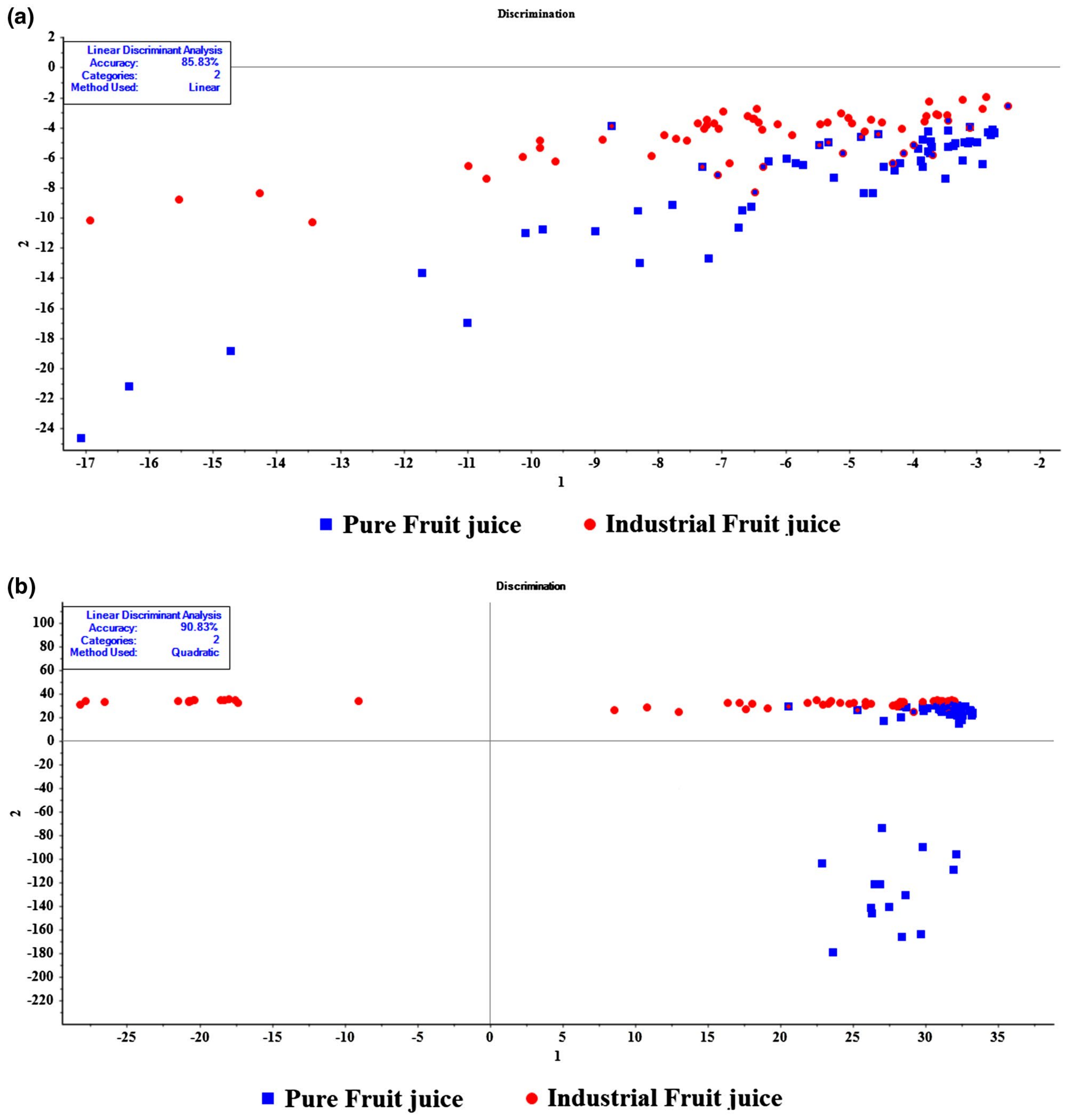

FIGURE 4 Classification of pure and industrial fruit juices using (a) LDA and (b) QDA methods

frequency response, leading to accuracies of 80.56% and 83.33%, respectively (Lashgari & MohammadiGol, 2016). In another study, an electronic nose was used with a chemometrics method to track the quality of tomato juice. The LDA method was more accurate than the QDA method, with 97.67% for the former and 82.33% for the latter (Hong et al., 2015).

TABLE 2 Confusion matrix of the fruit juices classification using LDA and QDA methods

Model	Type of fruit juice	Pure	Industrial
LDA	Pure	*54*	11
	Industrial	6	*49*
QDA	Pure	*55*	6
	Industrial	5	*54*

The italic values show of the correctly classified observations.

3.3 | SVM analysis

Two C/Nu-SVM methods were used to classify the fruit samples. The parameters Nu, C, and γ were validated by trial and error. Training and testing allocated 70% and 30% of the data to themselves, respectively. Table 6 shows the results of the SVM method for the prediction of the types of fruit juice based on two groups of pure and industrial fruit juice, and the classifications based on eight groups of fruit juice. The polynomial function had the highest

TABLE 3 Performance parameters of LDA and QDA models for classification of the fruit juices

Model	Type of fruit juice	Accuracy	Precision	Sensitivity	Specificity	AUC
LDA	Pure	0.919	0.831	0.900	0.926	0.913
	Industrial	0.971	0.891	1.000	0.963	0.981
Average per class		**0.945**	**0.861**	**0.950**	**0.944**	**0.947**
QDA	Pure	0.947	0.902	0.917	0.960	0.938
	Industrial	0.976	0.915	1.000	0.968	0.984
Average per class		**0.962**	**0.908**	**0.958**	**0.964**	**0.961**

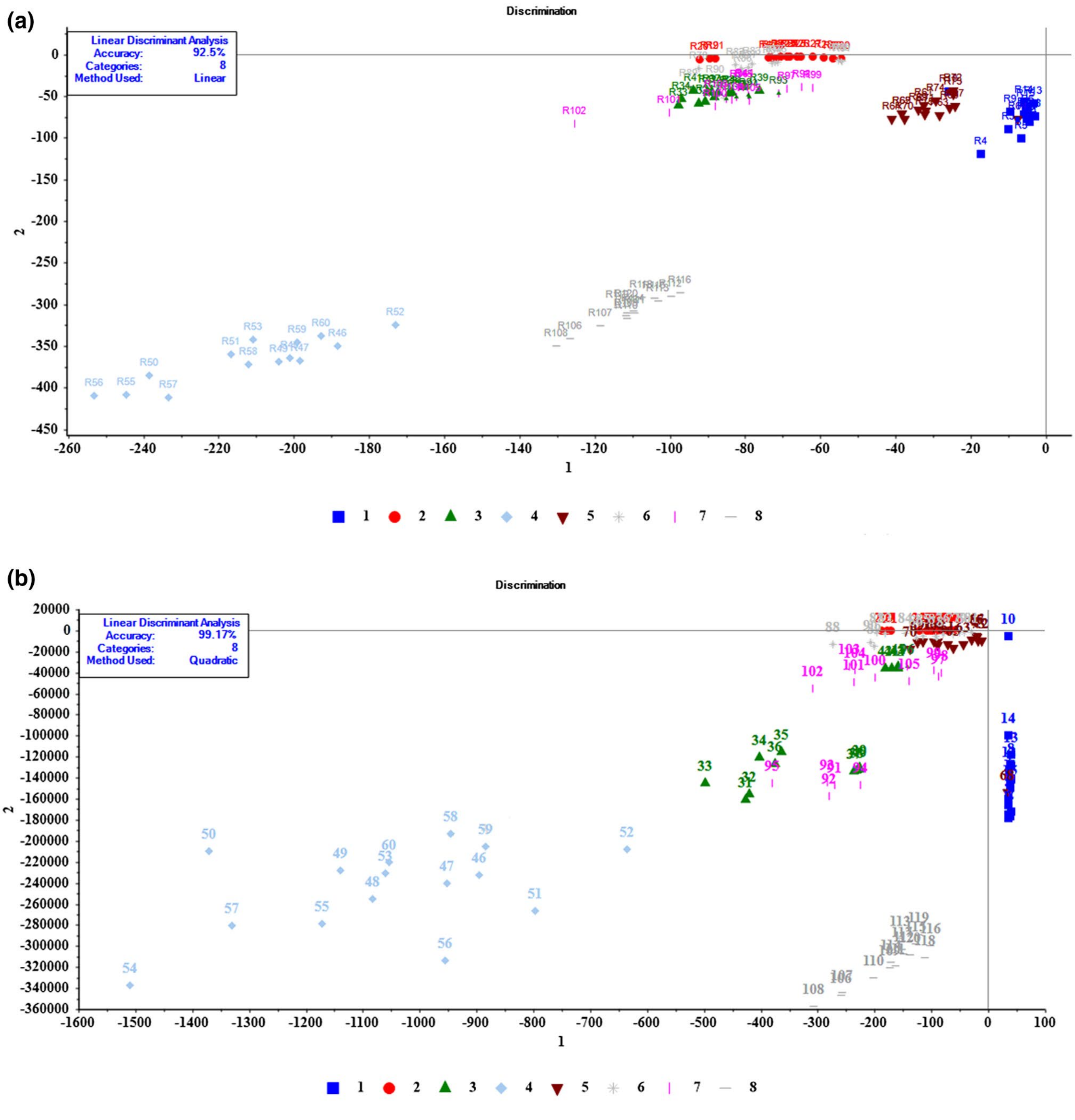

FIGURE 5 Classification all of type pure and industrial fruit juices using (a) LDA and (b) QDA methods

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TABLE 4 Confusion matrix of the types of fruit juices classification using LDA and QDA methods

Model	Type of fruit juice	M1	L1	O1	S1	M2	L2	O2	S2
LDA	M1	*14*	0	0	0	1	0	0	0
	L1	0	*15*	0	0	0	0	0	0
	O1	0	0	*13*	0	0	0	5	0
	S1	0	0	0	*15*	0	0	0	0
	M2	1	0	0	0	*14*	0	0	0
	L2	0	0	0	0	0	*15*	0	0
	O2	0	0	2	0	0	0	*10*	0
	S2	0	0	0	0	0	0	0	*15*
QDA	M1	*15*	0	0	0	0	0	0	0
	L1	0	*15*	0	0	0	0	0	0
	O1	0	0	*15*	0	0	0	1	0
	S1	0	0	0	*15*	0	0	0	0
	M2	0	0	0	0	*15*	0	0	0
	L2	0	0	0	0	0	*15*	0	0
	O2	0	0	0	0	0	0	*14*	0
	S2	0	0	0	0	0	0	0	*15*

The italic values show of the correctly classified observations.

TABLE 5 Performance parameters of LDA and QDA models for classification types of fruit juices

Model	Type of fruit juice	Accuracy	Precision	Sensitivity	Specificity	AUC
	M1	0.990	0.933	0.933	0.995	0.964
	L1	1.000	1.000	1.000	1.000	1.000
	O1	0.976	0.722	1.000	0.974	0.987
	S1	1.000	1.000	1.000	1.000	1.000
	M2	0.995	0.933	1.000	0.995	0.997
	L2	1.000	1.000	1.000	1.000	1.000
	O2	0.990	0.833	1.000	0.990	0.995
	S2	1.000	1.000	1.000	1.000	1.000
Average per class		**0.994**	**0.928**	**0.992**	**0.994**	**0.993**
	M1	1.000	1.000	1.000	1.000	1.000
	L1	1.000	1.000	1.000	1.000	1.000
	O1	0.995	0.938	1.000	0.995	0.997
	S1	1.000	1.000	1.000	1.000	1.000
	M2	1.000	1.000	1.000	1.000	1.000
	L2	1.000	1.000	1.000	1.000	1.000
	O2	1.000	1.000	1.000	1.000	1.000
	S2	1.000	1.000	1.000	1.000	1.000
Average per class		**0.999**	**0.992**	**1**	**0.999**	**0.999**

classification accuracy for eight groups of juices in the C-SVM method, so that the classification accuracy for training and validation data was 100% and 93.33%, respectively. However, the radial basis function had the highest accuracy in the Nu-SVM method, with values of 100% and 97.5% for the training and validation data, respectively.

In addition, another classification was performed for the two groups of pure and industrial fruit juices, based on which the linear function had the highest accuracy in both C-SVM and Nu-SVM methods. In general, the Nu-SVM and C-SVM methods were more suitable for the 8-group and 2-group classifications, respectively. In a study that used electronic nose combined with chemometrics

TABLE 6 Results and comparison of Nu-SVM and C-SVM models subjected to the kernel functions

	C-SVM				Nu-SVM			
Kernel function	*c*	*γ*	Train	Validation	Nu	*γ*	Train	Validation
Categories 8								
Linear	10	1	93.33	90.83	0.255	1	96.67	95.83
Polynomial	**100**	**100**	**100**	**93.33**	0.255	10	98.33	96.67
Radial basis function	100	100	99.17	98.33	**0.01**	**100**	**100**	**97.5**
sigmoid	100	1	92.5	90	0.255	1	97.5	95
Categories 2								
Linear	100	1	83.33	71.67	0.5	1	87.5	82.5
Polynomial	10	10	90	89.17	0.255	10	84.17	85.83
Radial basis function	**100**	**100**	**99.17**	**96.67**	**0.255**	**100**	**96.67**	**94.17**
sigmoid	100	0.1	74.17	70.83	0.5	0.1	74.17	75

Bold values show the highest classification accuracy.

method to track the quality of tomato juice, the SVM method had an accuracy of 94.17%, and the values of the best pairs of C and γ were 8 and 0.5, respectively (Hong et al., 2015). In another study, which aimed to describe the freshness of strawberries packed in polymer packages by the response surface method (RSM), the accuracy of sample classification by the SVM method through the polynomial kernel of the C-SVM function for training and validation was 86.4% and 50.6%, respectively. Also, the training accuracy and validation were 85.2% and 55.6%, respectively, for the Nu-SVM function using the radial basis (Ghasemi-Varnamkhasti et al., 2019). In addition, the linear function had the highest accuracy for detection of oxidation in edible oil using the electronic nose and SVM analysis. Accordingly, the accuracy for training and validation was 98%, 97%, 97%, and 95% for C-SVM and Nu-SVM, respectively (Karami et al., 2020; Karami Rasekh et al., 2020b). Timsorn et al. (2017), for identification of adulteration in uncooked Jasmine rice at room temperature and 55°C with RBF function, they achieved 100% classification accuracy. Similar results have been reported by other researchers (Huang et al., 2015; Tohidi et al., 2018).

4 | CONCLUSIONS

In this paper, an analytical method was developed for the evaluation of different types of fruit juice and detection of adulteration in pure and industrial juices, using an electronic nose along with data analysis. According to the results, the SVM method had higher accuracy compared to the QDA and LDA methods. Classification accuracy for SVM, QDA, and LDA methods was 96.25%, 95.8%, and 94.4%, respectively. The results of this study indicate that E-Nose, in combination with chemometrics methods, can be used to determine the quality of fruit juice with satisfactory results.

CONFLICT OF INTEREST

The authors declared that they have no conflict of interest.

AUTHOR CONTRIBUTIONS

Hamed Karami: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Resources; Software; Validation; Writing-original draft; Writing-review & editing. **Mansour Rasekh:** Funding acquisition; Project administration; Supervision; Visualization.

ETHICAL APPROVAL

This article does not contain any studies with human participants or animals performed by any of the authors.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

ORCID

Hamed Karami https://orcid.org/0000-0002-0654-6149

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