Detecting the adulteration in apple vinegar using olfactory machine coupled PCA and ANN methods

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Abstract: Nowadays, the number of food adulteration cases is increasing sharply for reasons such as population growth, increasing demand, and profitability of suppliers. Mixing apple vinegar with white vinegar and acetic acid is the most common method of cheating on the market in Iran. In this study, an electrical olfactory system was used to detect pure apple vinegar from acetic acid and white vinegar based on their odors. Acetic acid and white vinegar were added to pure apple vinegar at levels of 0-25-50-75 volume percent. An electronic nose system based on 8 metal oxide semiconductor sensors was evaluated to classify apple vinegar based on acetic acid and white vinegar adulteration. The data obtained from the sensors were analyzed by PCA and ANN methods after preprocessing. Based on the results, TGS822 and MQ136 sensors showed the highest response to the odor of samples of vinegar mixed with acetic acid and white vinegar, respectively. Also, the confusion matrix obtained from ANN analysis for different levels of adulteration with acetic acid and white vinegar showed a correct classification rate of 93.3% and 94.7%, respectively.

Keywords: electronic nose, apple vinegar, white vinegar, acetic acid, adulteration.

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1 Introduction

Nowadays, the health of people and the progress of society are directly related to the health and desirability of food quality (Taheri Gravand et al., 2020). Adulteration in the field of food production has a historical background and is not specific to today. However, due to increasing demand is becoming more than ever. Methods of food adulteration have become increasingly sophisticated and sometimes surpassing advances in decomposition science (Karami et al., 2020). In addition to affecting product quality and financial losses, these adulterations are also harmful to consumers' health, thus causing concern and loss of consumer confidence (Kiani et al., 2016).

Vinegar is a natural product and one of the most important components of the Mediterranean diet, which is obtained from the fermentation process that is mainly used in food preparation due to its taste and smell, and is one of the most famous anti-infective drugs. Apple turns into vinegar when fermented, and because they have already been completely crushed for this purpose, it does not lose its nutritional properties at all, and only its sugar is converted to acid (vinegar), which contains minerals such as potassium,

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chlorine, sodium, magnesium, lime, sulfur, iron, fluorine, silicon, etc. (Samad et al., 2016).

It is very difficult to distinguish artificial and low-quality vinegar due to their color, smell, and taste similar to natural vinegar, and so far, no scientific method has been introduced for this, except for traditional methods which are not very reliable. It was, therefore, important to look for an easy, fast, and low-cost method to do this. The olfactory machine is one of the new methods of examining and determining the quality of food. This device simulates the human olfactory sense and compares it with the existing odor above the samples by means of a sensor array of non-specific sensors (Ghasemi-Varnamkhasti et al., 2015). Detection of complex odors with the help of chemical and electronic sensors, by simulating the human sense of smell, is the most basic work of the electronic nose system (Ayari et al., 2018a).

Given that adulteration in milk and other dairy products is a serious threat to human health and leads to numerous economic losses, in a study, Tohidi et al. (2018), developed an electronic nose system in combination with pattern recognition methods. They detected formalin adulteration in raw milk and concluded that the olfactory machine based on metal oxide semiconductor (MOS) sensors has the ability to detect formalin adulteration in raw milk.

The use of electronic nose is also widely used in various other fields such as essential oil detection. Gorgi-Chakespari et al. (2017), studied the performance of an electronic nose system in the qualitative classification of rosemary essential oil with the help of artificial intelligence. Khodamoradi (2020), in a study investigated the performance of an electronic nose system for classifying basil and savory plants based on the amount of applied urea fertilizer during plant breeding. The results showed that the sensors showed different reactions to the aroma of basil and savory. In another research, an electronic nose system was used to detect fresh chicken meat from the frozen type (Taheri Gravand et al., 2020).

White vinegar and acetic acid are used in the market as common adulterations in original apple vinegar in Iran. Due to the disadvantages of using counterfeit vinegar and lacking the properties of natural vinegar, it is necessary to find a safe, simple and inexpensive way to detect adulteration vinegar. Therefore, the aim of this study was to investigate the use of electronic nose technology for detecting pure apple vinegar of adulterated ones (mixed with acetic acid and white vinegar).

2 Materials and methods

2.1 Preparation of the samples

For experiments, pure apple vinegar was purchased from a reputable market which is producer of vinegar in Kermanshah city (located in the west of Iran with a Longitude of 47.0777685 and Latitude of 34.3276924). Also, white vinegar and acetic acid (Merck 100063.2500) were purchased. One day before the experiment with the electronic nose, white vinegar, and acetic acid were added to pure apple vinegar at levels of 0-25-50-75 volume percent. For each sample, the experiments were performed with 15 replications (in the Physical properties laboratory, mechanical engineering of biosystems department, Razi University, Kermanshah, Iran).

2.2 Electronic nose system

The olfactory machine is an advanced analytical tool that is a good alternative to conventional methods which are used in the food industry. Estimating the concentration or determining some intrinsic properties of a fragrant compound is something that the olfactory machine can easily do, something that the human nose can hardly do. But olfactory machines do not provide any information about odor-forming compounds and their other characteristics (Ghasemi-Varnamkhasti et al., 2011). The used olfactory machine system has 8 metal oxide semiconductor sensors (MOS) including MQ136, TGS822, MQ9, MQ3, TGS813, TG2620, TG2602, and MQ135, which each of them reacts to specific combinations of volatiles in the samples. These sensors are widely used in the olfactory machine due to their high chemical stability, long life, low response to moisture and reasonable price (Ayari et al., 2018b). Figure 1 shows the actual schematic of used the electronic nose system.

Figure 1 The used electronic nose system

The used system has two parts including hardware and software. The hardware part includes: a data acquisition system, sensors, sensor chamber, sampling chamber, power supply, fittings and accessories, electric valves, air pump, and air filter (Ayari, 2017). The software section also included LABVIEW 2012 software. Figure 2 shows the working steps of an electronic nose in the form of a block diagram.

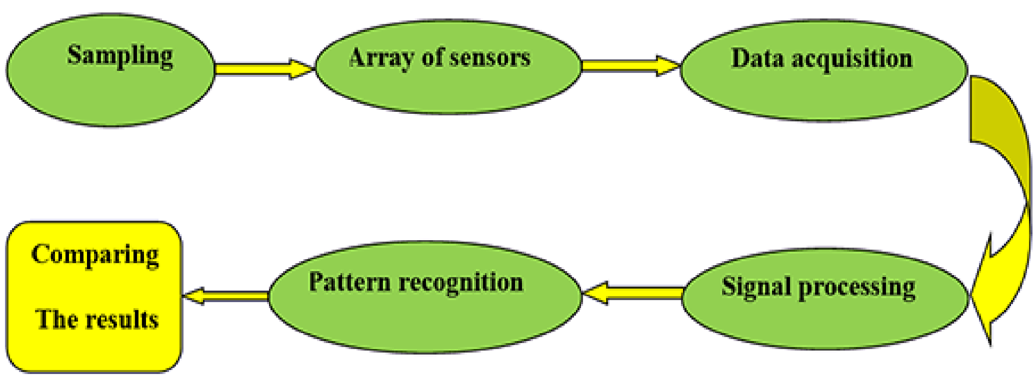


Figure 2 The flowcharts of the electronic nose working steps

2.3 Data collection process

The static head space method, which is the most common and cheapest data collection method, was used in this study. At the first, 30 grams of the sample was put into the sample chamber and the lid was closed. In order to fill the air inside the chamber with the sample odor and fill the space above the sample, the operator waited about 15 minutes and after that the sampling data was started. The purpose of this operation was to saturate the air inside the chamber with the sample odor and increase the concentration of volatile substances inside the chamber (Tohidi et al., 2018).

Sampling data were performed by the olfactory machine in three stages: baseline correction, sample odor injection, and sensor cleaning. At the baseline stage, clean air was passed over the sensor to bring the sensor array response to a stable state for 200 seconds. During the injection, with the entry of gas around the sample into the sensor chamber for 150 seconds, a change was made in the output voltage of each sensor (according to the type of sensor and the degree of sensitivity). During the cleaning phase of the sensors and the chamber, the clean air was passed over the sensors in order to bring the response of the sensor array to a stable state. Also, at this stage, the pump would remove the odor left inside the sample chamber. This step also took 150 seconds. Thus, the system was prepared for the next experiment (Ayari et al., 2018a; Sanaeefar et al., 2014). The voltage response of the sensors during these 500 seconds is collected by the data acquisition system. Figure 3 shows an example of the sensors' response to the sample odor at these times.

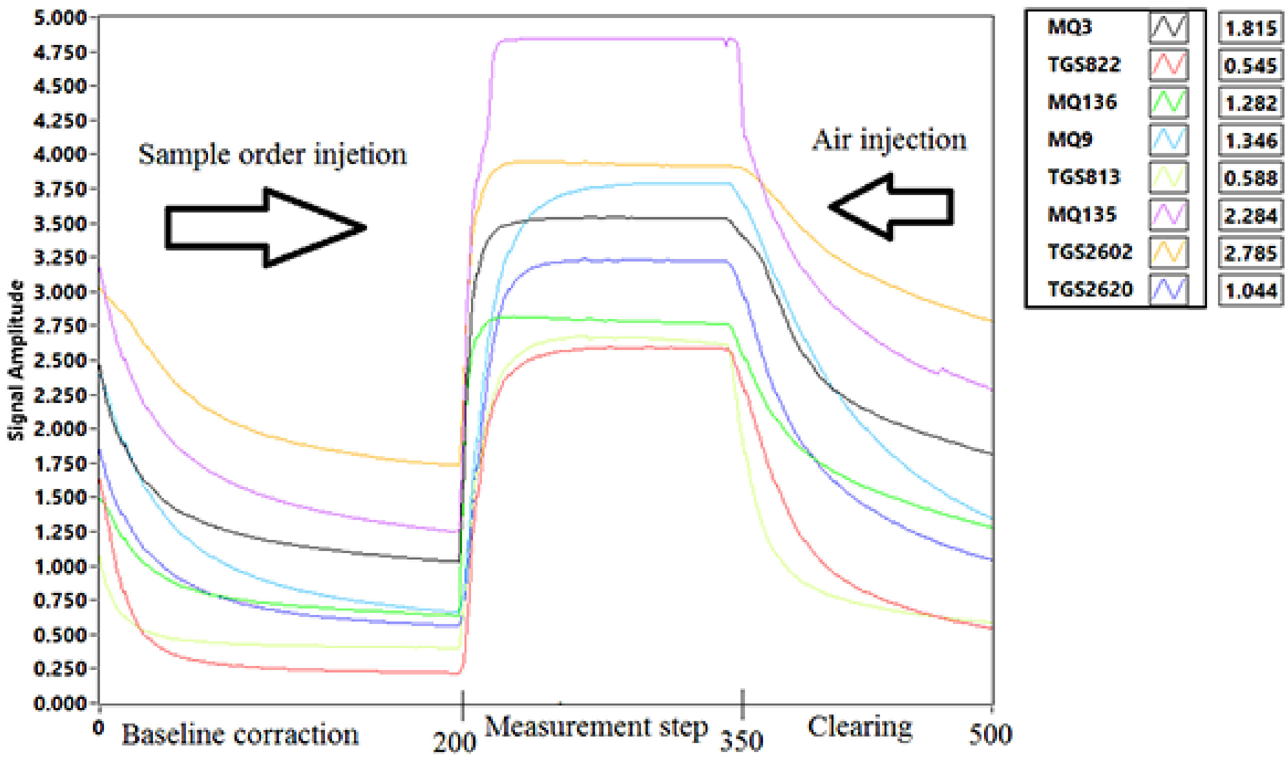


Figure 3 An example of the response of sensors to the sample odor in LabView2012 software

2.4 Data analysis

The data obtained from the sensors were first stored as raw data. In the next step, the sensor signals were pre-processed. The first step in analyzing the data is to pre-process the signal of each sensor. Pre-preprocessing has a great impact on the performance of pattern recognition methods and also depends to some extent on the type of sensors and is usually different. Signal pre-processing is possible with three methods: differential, relative and fractional. In this study, data were pre-processed by the fractional method. In the fractional method, the baseline is subtracted from the sensor response and then divided by the baseline. The obtained response is not only dimensionless but also normalized and can be used for small or large signals (Arshak et al., 2004):

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

Where $Y_s(t)$ is the normalized response, $X_s(0)$ is the baseline, and $X_s(t)$ is the sensor response. The pre-processed data were used as input data for principal component analysis (PCA) and artificial neural network (ANN) analytical methods. The PCA method is an unsupervised multivariate analysis method that is used to reduce the size of the data (Li et al., 2007). The PCA method is widely used to identify patterns and classifications of data and to express the data in a way that makes the similarities and differences between them clearer (Mahmoudi, 2009). The PCA analysis was done using the Unscrambler X 10.4 software.

To classify the samples based on the degree of adulteration, the ANN method (ANN) was used. The network training algorithm, the Lunberg-Marquardt method, and the hyperbolic tangent activation function were used for the hidden and output layers, respectively. The performance of the designed networks was evaluated using mean square error (MSE) and correlation coefficient (R). In this study, 75% of the data was used for training, 10% for validation, and 15% for testing (Khodamoradi et al., 2020). Statistical indices which were derived from confusion matrices such as accuracy (*Ac*), sensitivity (*Se*), specificity (*Sp*), precision (*Pr*), and area under curve (*AUC*) were also used to evaluate the proposed classification system (Sokolova and Lapalme, 2009; Basri et al., 2017; Mahmodi et al., 2019):

$$Ac = \frac{TP + TN}{TP + TN + FP + FN} \times 100\% \tag{2}$$

$$Se = \frac{TP}{TP + FN} \times 100\% \tag{3}$$

$$Sp = \frac{TN}{TN + FP} \times 100\% \tag{4}$$

$$Pr = \frac{TP}{TP + FP} \times 100\% \tag{5}$$

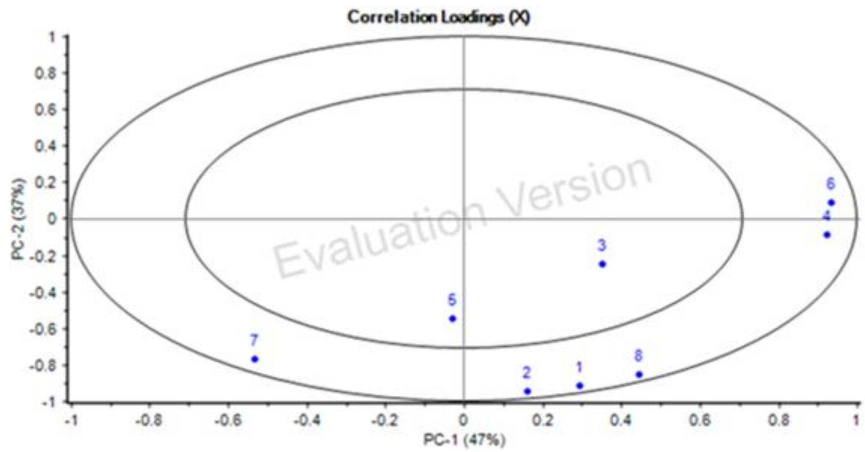
$$AUC = \frac{1}{2}\left(\frac{TP}{TP + FN} + \frac{TN}{TN + FP}\right) \times 100\% \tag{6}$$

In these relationships, *TP* is positive correct decisions, *FP* is positive incorrect decisions, *TN* is negative correct decisions and *FN* is negative incorrect decisions. In fact, *TP* and *TN* show the correct predictions of the model, and *FP* and *FN* show the wrong predictions of the model. Accuracy focuses on the overall effect of the classifier. Precision evaluates the condition of the data label class with the positive tags specified by the classifier. The sensitivity recognizes the effect of the classifier on positive labels and how the effect of the classifier with a negative label. Finally, the area under curve shows the ability of the classifier to avoid incorrect classification (Taheri Gravand et al., 2020). The analysis of ANN was performed by MATLAB 2014b software.

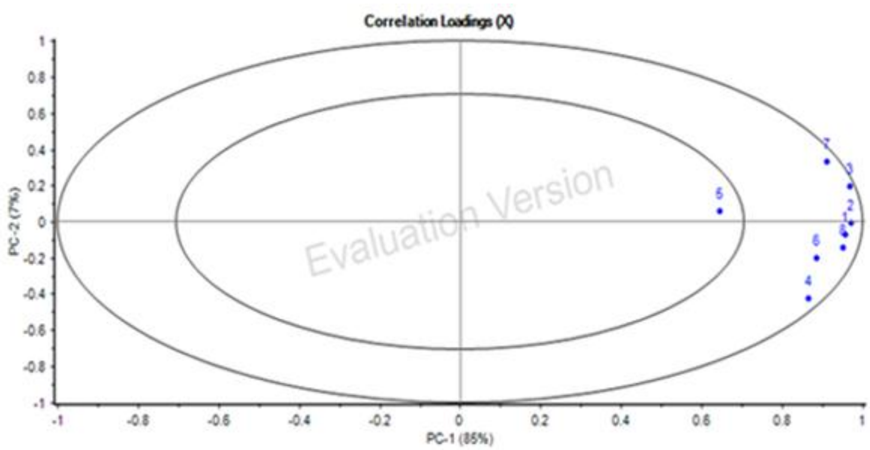
3 Results and discussion

3.1 PCA Analysis

PCA is a common method for visual analysis and classification (Yin et al., 2013). In this study, this pattern recognition method was used to reduce the dimensions of pure apple vinegar with different percentages of adulteration of acetic acid and white vinegar by performing dimensional reduction from eight variables to two or three main components and maintaining the most basic information contained in the data set. Based on the results of PCA analysis in acetic acid adulteration, the two main components of PC-1 and PC-2 were 47% and 37%, respectively, and the variance between the samples was totally 84%. Figure 4a (loading plot) shows PCA analysis for a mixture of apple vinegar with acetic acid. Based on the results, the highest value on the principal component is obtained for sensor number 2 (TGS822), which is mostly used in the detection of organic solvents. It must be said that the sensors which have a low loading coefficient or similar values have no or similar effect on pattern recognition and can be removed from the olfactory system to lower the final cost (Ayari et al., 2018a). The loading diagram for PCA analysis results for a mixture of apple vinegar with white vinegar is given in Figure 4b. As shown in Figure 4b, in the adulteration of apple vinegar with white vinegar, the two main components PC-1 and PC-2 separated 85% and 7% of the samples, respectively, and the variance between the samples was 92% of the total data. The results showed that sensor number 3 (MQ136) showed the highest reaction to the tested samples (related to the mixture of apple vinegar with white vinegar). This sensor mostly reacts to compounds containing sulfur dioxide. Tohidi et al. (2018), reported that the MQ4, TGS822, and TGS2620 sensors were the best sensors for detecting milk adulteration using a loading diagram. In another research, Ayari et al. (2018a), introduced the TGS822 sensor as the best sensor for detecting the level of cheating in pure cow ghee. In similar studies, Sanaeefar et al. (2014), to identify the stages of banana ripening, and Khodamoradi et al. (2020), to detect the level of applied urea fertilizer in savory, presented the best sensor using the loading plot.



(a) Loading plot of PCA analysis related to adulteration pure apple vinegar with acetic acid



(b) Loading plot related to adulteration pure apple vinegar with white vinegar

Figure 4 Loading plot results of PCA analysis

3.2 Classification results with ANN

3.2.1 Adulteration with acetic acid

In this study, in order to classify and detect acetic acid adulteration in pure apple vinegar, an ANN algorithm was used. The results of the analysis of ANN for samples mixed with acetic acid are given in Table 1. Based on the results, the best neural network model had an 8-5-5 structure. This structure has 8 neurons in the input layer (number of sensors), a hidden layer with 5 neurons, and 5 neurons in the output layer (5 different classes). The values of correlation coefficient (R) and mean square error (MSE) for the best structure were 0.995 and 1.41×10^{-2}, respectively. The confusion matrix for this structure is also given in Table 2. As it can be seen, the correct classification rate for detecting acetic acid adulteration in pure apple vinegar is 93.3%. It must be said that, the olfactory machine has a good track record in terms of classification. In this way, the analysis of the ANN was able to separate the samples with high accuracy.

Table 1 The results of ANN analysis for pure apple vinegar adulteration with different levels of acetic acid

Model structure	Correlation coefficient (R)	Mean square error (MSE)
8-3-5	0.627	8.59×10^{-2}
8-4-5	0.808	5.19×10^{-2}
8-5-5	**0.955**	$\mathbf{1.41\times10^{-2}}$
8-6-5	0.789	5.73×10^{-2}
8-7-5	0.862	4.03×10^{-2}

Table 2 Confusion matrix obtained to identify the pure apple vinegar from acetic acid adulteration

Samples	1 pure acetic acid	2 25% adulteration	3 50% adulteration	4 75% adulteration	5 pure apple vinegar
1	15	0	0	0	0
2	0	14	0	1	0
3	0	0	12	0	0
4	0	0	0	14	0
5	0	0	0	0	15
Correct classification rate: 93.3%					

Using the confusion matrix, the values of statistical indices for 5 classes were calculated for the test data (Table 3). According to Table 3, the mean values of statistical indicators including accuracy, precision, sensitivity, specificity, and area under curve were equal to 97.3%, 93.8%, 93.3%, 98.3%, and 95.8%, respectively. As can be seen from the results of Tables 2 and 3, the ANN classifier was able to detect the levels of acetic acid adulteration in pure apple vinegar.

In another research, an evaluation of the performance of an electronic nose system for the classification of rosemary essential oil using a multilayer perceptron neural network was used. Training and test data showed 100% and 96% classification accuracy, respectively (Gorgi-Chakespari et al., 2017).

Table 3 Performance parameters of 8-5-5 ANN classifier for test data (acetic acid adulteration)

Class	Accuracy	Precision	Sensitivity	Specificity	AUC
1	100	100	100	100	100
2	97.3	93.9	93.3	98.3	95.8
3	96	100	80	100	90
4	94.6	82.3	93.3	95	94.1
5	98.6	93.7	100	98.3	99.1
Average per class (%)	**97.3**	**93.8**	**93.3**	**98.3**	**95.8**

3.2.2 Adulteration with white vinegar

The results of the analysis of ANN for samples mixed with white vinegar are given in Table 4. Based on the results, the best neural network model has a structure of 8-6-5. This structure has 8 neurons in the input layer (number of sensors), a hidden layer with 6 neurons and 5 neurons in the output layer (5 different groups). The values of correlation coefficient (R) and mean square error (MSE) for the best structure were 0.937 and 1.9×10^{-2}, respectively. The confusion matrix for this structure is also given in Table 5. It can be seen that, the correct classification rate for detecting the amount of white vinegar adulteration in pure apple vinegar was 94.7%. The values of statistical indices for 5 data groups were calculated for the test data using the confusion matrix (Table 6). According to Table 6, the mean values of statistical indicators including accuracy, precision, sensitivity, specificity, and area under curve were equal to 97.86%, 94.39%, 94.66%, 98.66%, and 96.66%, respectively. From the results, it is clear that the ANN algorithm was able to detect the adulteration rate of white vinegar in pure apple vinegar. In research, the detection of adulteration in edible oil using the olfactory system and analysis of ANN was evaluated. Based on the results, the ANN was able to detect the rate of adulteration in fresh edible oils with an accuracy of 97.3% (Karami et al., 2020). In similar studies, the combination of the olfactory machine method and the ANN was used to detect adulteration in saffron (Heidarbeigi et al., 2015) and animal caw oil (Ayari et al., 2018b).

Table 4 The results of ANN analysis for apple vinegar adulteration with different levels of white vinegar

Model structure	Correlation coefficient (R)	Mean square error (MSE)
8-4-5	0.897	3.71×10^{-2}
8-5-5	0.823	7.49×10^{-2}
8-6-5	**0.937**	$\mathbf{1.9\times10^{-2}}$
8-7-5	0.919	2.62×10^{-2}
8-8-5	0.915	2.69×10^{-2}

Table 5 Confusion matrix obtained to identify the pure apple vinegar from white vinegar adulteration

Samples	1 pure white vinegar	2 25% adulteration	3 50% adulteration	4 75% adulteration	5 pure apple vinegar
1	15	0	0	0	0
2	0	12	1	1	0
3	0	0	14	0	0
4	0	0	0	15	0
5	0	0	0	0	15

Correct classification rate: 94.7%

Table 6 Performance parameters of 8-6-5 ANN classifier for test data (white vinegar adulteration)

Class	Accuracy	Precision	Sensitivity	Specificity	AUC
1	100	100	100	100	100
2	94.66	92.30	80	98.33	98.16
3	94.66	82.35	93.33	95	94.16
4	100	100	100	100	100
5	100	100	100	100	100
Average per class (%)	97.86	94.39	94.66	98.66	96.66

4 Conclusion

In this study, an electronic nose system based on 8 metal oxide semiconductor sensors was evaluated to classify apple vinegar based on acetic acid and white vinegar adulteration. PCA and ANN analysis were used to evaluate the ability of the olfactory system in classification. The results of PCA analysis identified TGS822 and MQ136 sensors as the best sensors for detecting acetic acid and white vinegar adulteration, respectively. The results also showed that the main components of PC1 and PC2, for samples mixed with acetic acid and white vinegar, covered 84% and 92% of the variance of the data, respectively. Also, the confusion matrix of the best ANN structure for cheating with acetic acid and white vinegar showed a correct classification rate of 93.3% and 94.7%, respectively. The results of the research showed that this system can be used in the field of adulteration in apple vinegar.

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