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Identification of olfactory characteristics of edible oil during storage period using metal oxide semiconductor sensor signals and ANN methods

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Abstract

In this study, an electronic nose coupled with artificial neural network (ANN) was used to predict the shelf life of two oil with new production date and oil with old production date over a period of 150 days. According to the American Oil Chemists' Society results, the oils were oxidized after 60 days. Principal component analysis results indicated that all the oil samples were correctly discriminated from each other during their storage times, and samples of oxidized and nonoxidized oils can be properly distinguished from each other. Two main components (PC1, PC2) managed to describe 97% of the data set variance concerning the shelf life of the oil. To develop the ANN models, the data were first divided into three groups: training (60%), validation, and test (40%). To determine the best model, two criteria (R^2 and root mean square error) were used. The results revealed that the ANN model can be used as a powerful tool for pattern recognition and determination of the shelf life of oil and its oxidation degree at high precision. Scientific and feasible results can be obtained by matching ANN and the results obtained by metal oxide semiconductor sensors of E-nose.

Practical applications

One of the most important causes of food spoilage is lipid oxidation. The American Oil Chemists' Society (AOCS) has developed a variety of methods for assessing the state of food oxidation. In this study, oil shelf life studied by a combination of artificial senses and chemometrics methods. The acidity, peroxide, anisidine, and Totox values of the edible oil samples were measured according to the AOCS standard. Principal component analysis and artificial neural neural methods succeeded in classifying the samples based on their storage time with high accuracy.

1 | **INTRODUCTION**

In recent decades, industrialization and modernization of the communities have decreased the daily activities of the people; thus, physicians are increasingly recommended the use of low-cholesterol vegetable oil. The nutritional value of the vegetable oils is incomparable with the animal-based or hydrogenated vegetable oils. Liquid vegetable oils obtained from sesame, olive, corn, soybean, and sunflower are among the best edible oils for human health. These oils

contain considerable amounts of unsaturated fatty acids, which are vital for human health as they reduce the chance of hyperglycemia, atherosclerosis, and other cardiovascular diseases. Therefore, a vegetable oil obtained from the oilseeds is the best edible oil.

Vegetable oils are the main source of unsaturated fatty acids and other substances, for example, phytosterols and tocopherols in the human diet. Oxidation is an important chemical reaction that can affect the nutritional value and sensory quality of vegetable oils. Fatty acids are the main compounds of oil that undergo oxidation

FIGURE 1 Schematic of an artificial olfactory (E-nose) system. The components of this system consist of the following parts: (a) air filter (activated charcoal to remove VOC hydrocarbons), (b) sample headspace chamber, (c) solenoid air valves, (d) diaphragm pump, (e) E-nose sensor array chamber, (f) data acquisition card, (g) personal computer, and (h) air outlet line from sensor array chamber (for exhaust gases)

because they have unsaturated bonds in their structure. Other unsaturated compounds in oils, such as phytosterols and tocopherols, are also sensitive to oxidation (Karami et al., 2020a, 2020c; Kmiecik et al., 2019).

The quality of the oil depends on its resistance to oxidation. Oxidation of oil may cause loss of bioactive compounds and nutrients and formation of toxic compounds (Castelo-Branco et al., 2016). Parameters that increase oil oxidation include long-term storage in adverse environmental conditions such as light exposure, high temperature, or oxygen. Moreover, oxidation is one of the main factors reducing edible oil quality and often determines its shelf life (Wang et al., 2019).

Oxidation is observed during oil storage and in the frying process/oil heating. In addition, oxidation occurs in oils under the influence of oxygen from the air. However, hydrolysis and thermal changes are also observed in oil exposed to humidity and high temperatures in the fried product. Three reactions in heated oil give rise to two new components; the first group of compounds with lower molecular weight, compared with parent triacylglycerols, comprises oxidized fatty acids, hydrocarbons, ketones, aldehydes, epoxides, or alcohols. The second group consists of compounds with higher molecular weight than that of parent triacylglycerols, that is, trimers, oligomers of triacylglycerols, and dimmers. The third group of compounds, that is, volatile substances, has a considerable impact on the sensory characteristics and quality of fried food and heated oil (Karami et al., 2020a, 2020c; Rusinek et al., 2021).

American Oil Chemists' Society (AOCS) has developed various indicators to assess the state of oil oxidation, such as the anisidine index (AnV), the acidity index (AV), the peroxide index (PV), and the Totox index. These chemical tests are not difficult, but they are timeconsuming and destructive. They also pose potential hazards to the environment and human health due to solvent wastes (Karami et al., 2020b, 2020c).

Therefore, the most evident approach is to rely on an automatic system such as an E-nose, which not only mimics the human olfaction but also is capable of detecting and classifying the toxic vapors through a complicated method. Some of the toxic compounds can be easily identified by an E-nose (Srivastava et al., 2019).

Using the olfactory machine, the durability of various products such as tomatoes (Gómez et al., 2008), apples (Brezmes et al., 2001; Saevels et al., 2004), raw milk and meat (Amari et al., 2009), *Valerianella* (Cortellino et al., 2018), fried potatoes (Chatterjee et al., 2014), rice (Malegori et al., 2020), essential oils from herbs and fruits (Rasekh et al., 2021), and agricultural products (Baietto & Wilson, 2015; Gancarz et al., 2021; Marek et al., 2020; Wilson, 2013) has been determined.

Pattern recognition techniques and data analysis are required to detect the signals or their patterns to identify and classify the data. The E-nose-obtained signal pattern can be analyzed using ANN and statistical tools such as discriminant factorial analysis, ANN, and principal component analysis (PCA; Srivastava et al., 2019).

In this context, the present study is aimed at the predicting of the shelf life of edible oils using a combination of E-nose, artificial neural network (ANN) and PCA. The results of this research were confirmed by comparison with the AOCS methods. In this regard, the development of a proper and powerful classification model capable of offering satisfactory results (of edible oils shelf life) under real conditions sounds crucial. This method can be useful as a simple and cost-effective method to control the quality of oils during their storage period.

2 | **METHODS**

Edible oils (which is a combination of three oils: soybean, sunflower, and canola) oil with new production date (ONPD) and oil with old production date (OOPD) was prepared. The samples were stored at room temperature. Twenty milliliters of each oil was poured into a 50-ml sample container. In total, 40 oil samples with ONPD and OOPD were tested.

TABLE 1 Analysis of variance for the chemical parameters of edible oil

	Sources	Degrees of freedom	Mean square
p-Anisidine value	Factor A	$\mathbf{1}$	88.988
	Factor B	5	0.571
	AB	5	0.368^{*}
	Error	24	0.071
	Total	35	
Peroxide value	Factor A	$\mathbf{1}$	0.034
	Factor B	5	9.682 [*]
	AB	5	0.052 ^{ns}
	Error	24	0.020
	Total	35	
Acetic acid value	Factor A	$\mathbf{1}$	0.314
	Factor B	5	0.120
	AB	5	0.122 [*]
	Error	24	0.000
	Total	35	
Totox value	Factor A	$\mathbf{1}$	82.204
	Factor B	5	34.534
	AB	5	0.697 [*]
	Error	24	0.104
	Total	35	

Note: ns, non significant.

*Significant at *p* ≤ .01.

The olfactory machine used consisted of electronic components, eight sensors, pumps, electronic valves, air filters, and a software section for data analysis (Figure 1; Ayari et al., 2018b).

Data were collected from oil samples in three phases of cleaning, measuring, and cleaning the sensors, which took a total time of 550 s. In the first phase, the air was pumped into the sensor chamber for 200 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 150 s, which was long enough for the sensors to reach stable signal values. 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. The sensor array included eight metal oxide semiconductor sensors. MQ3 (alcohol), TGS822 (organic solvent steam), MQ136 (sulfur dioxide), MQ9 (carbon monoxide), TGS813 (methane, propane, and butane), MQ135 (ammonia, benzene, and sulfide steams), TGS2602 (hydrogen sulfide, ammonia, and toluene), and TGS2620

(alcohol and organic solvent steam) (Karami et al., 2020a, 2020b, 2020c).

The computer stored the output signal data of the sensors during the measurement period, as one datum per second. After the measurement process was complete, the obtained data were stored for 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 et al., 2020a, 2020b):

$$
Y_{s}(t) = \frac{X_{s}(t) - X_{s}(0)}{X_{s}(0)}
$$
\n(1)

where, *Ys* (*t*), *Xs* (0), and *Xs* (t) indicate the normalized sensor response, the baseline, and the sensor response, respectively.

Then, the PCA method was used to determine the shelf life of the edible oils using the obtained data. The data obtained from the mentioned processing (fraction method) were applied as the input. PCA is a multivariable, nonsupervised method for compacting the linear data and feature extraction. PCA can be also used to decrease the data dimension; in a way that it preserved those components of the data set with the highest impact on the variance. This method has been extensively used to demonstrate the E-nose responses to simple and complex smells offering qualitative data for pattern recognition. To determine the shelf life of the edible oil samples, the PCA method was used through Unscrambler x10.4.

Application of the ANN is an innovative method to solve various engineering problems. This method relies on finding the intrinsic relationship between the effective parameters of the problem, learning and then their generalization to similar samples. ANN is one of the most common methods of artificial intelligence (AI), which was first presented in the 60 s . The multilayer perceptron is one of the most applied ANN methods. This method often involves three layers such as input, output, and hidden layers. The independent and dependent variables are placed in the input and output layers, respectively. Generally, the ANN structure is composed of a group of computing units known as neurons or nodes. Each layer can encompass several neurons. The units of the input layer are only responsible for distributing the input values to the subsequent layers; thus, they do not conduct any computations (Haykin, 1998). Theoretically, there is no limitation for the number of hidden layers and their nodes; they can be determined by trial and error. Equations 7–11 can be used to determine the number of nodes in the hidden layer (Amari et al., 2009):

$$
\leq 2 \times N_i + 1 \tag{2}
$$

$$
\left(N_i + N_o\right)/2\tag{3}
$$

$$
2N_i/3 \tag{4}
$$

$$
\sqrt{\left(N_i \times N_o\right)}\tag{5}
$$

$$
2N_i \tag{6}
$$

FIGURE 2 Result of Duncan mean comparison test

FIGURE 3 Score plot of principal component analysis for predicting the shelf life of the oil

In the above equations, the number of the hidden layer nodes can be calculated from the number of nodes in the input (Ni) and output (NO) layers. The hyperbolic tangent activation function was used for the latent layer. The number of output layer neurons was selected based on the experiment type. According to three different experiments performed, initially, the data of two oils (ONPD and OOPD) in six periods were used for the shelf life of the oil, that is, the output layer had 12 neurons to evaluate the shelf life of the oil.

TABLE 2 Artificial neural network results

Bold parts show the best neural network structure for classification oils.

The next test was to identify healthy ONPD and OOPD (i.e., the first three periods when the oil has not yet been oxidized); because their production dates (PDs) were different, they were tried to be separated from each other; therefore, for this experiment, the output layer had six neurons. And in the next step, the only purpose was to detect nonoxidized and oxidized oils, regardless of their PD, so two neurons were considered for this experiment.

Accordingly, training, validation, and experiment allocated 60%, 20%, and 20% to themselves, respectively. The neural network training principally is based on trial and error. Efficiency evaluation is necessary after the training process of the neural network with proper structure. The optimal topology for the mentioned neural network is the highest value for the coefficient of determination of R^2 and the lowest for root mean square error (RMSE). The model input included data obtained from the response of eight sensors, so eight input layers were considered for the experiments. 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):

Sensitivity =
$$
\frac{TP}{TP + FN}
$$
 (7)

$$
Specificity = \frac{TN}{TN + FP}
$$
 (8)

$$
Precision = \frac{TP}{TP + FP}
$$
 (9)

$$
Accuracy = \frac{TP + TN}{TP + TN + FN + FP}
$$
 (10)

$$
AUC = \frac{\text{Sensitivity} + \text{Precision}}{2} \tag{11}
$$

where, TP (true positive), TN (true negative), FP (false positive), and FN (false negative) are indicated, and all values are dimensionless.

In this study, supervised learning algorithm was used for training and softmax activation function for the output layer. After training the ANN, its performance has to be investigated. For this purpose, RMSE and coefficient of determination (R^2) were used for fitting the predicted samples to the real ones. To this end, 60%, 20%, and 20% of the data were used for training, validation, and test, respectively.

FIGURE 4 Confusion matrix obtained for (a) shelf life, (b) detection of nonoxidized oils in six groups, and (c) classification of oxidized and nonoxidized oils

 | KARAMI et al. **7 of 10**

TABLE 3 Performance parameters of artificial neural network models

ANN is based on trial and error to find the best network configuration through varying the number of hidden layers and their neurons, activity functions, training algorithm, and the number of iterations in the training stage to lead to the intended output parameter. After training the ANN, its performance has to be investigated. The best network is the one with the highest R^2 and the lowest RMSE:

$$
R^{2} = 1 - \left[\sum_{1}^{n} \left(\frac{X_{pi} - X_{ei}}{X_{pi} - X} \right)^{2} \right]
$$
 (12)

RMSE =
$$
\sqrt{\frac{1}{n} \sum_{1}^{n} (X_{pi} - X_{ei})^2}
$$
 (13)

where, X_{pi} and X_{ei} are the predicted and observed values, respectively, *X* shows the mean values, and *n* denotes the number of data.

The parameters of acidity, peroxide, and anisidine were measured using AOCS method. Equation (14) can be used to calculate the Totox index (Hai & Wang, 2006):

$$
Totox = 2 \times (PV) + AnV \tag{14}
$$

The statistical analysis was conducted using a completely randomized factorial test.

3 | **EXPERIMENTS AND DISCUSSIONS**

3.1 | **Chemical analysis of oil**

After measurement by olfactory machine, a sample was measured by AOCS methods. The first step in identifying the primary stages of oxidation is to determine the peroxide index. The average peroxide for the oils on the first day was about 3 and after 150 days, it reached 6.5. Because peroxide is not a reliable indicator of oxidation, so the Totox index was used (Equation 14) (Billek et al., 1978).

Factorial experiment was performed with two factors of storage time and oil type. The type of oil (ONPD and OOPD) and storage time (150 days) and the tests were evaluated at 30-day intervals.

The results of analysis of variance of oils for Totox, peroxide, acidity, and anisidine indices are listed in Table 1. The effect of factor A (oil type) and factor B (time) as well as interaction (AB) was significant for Totox, acidity, and anisidine (at the *p* value of 1%), although factor A was not significant for the PV index. Figure 2 shows the comparison of mean values using the Duncan Multirange Comparison Test at the 1% probability level.

According to the results obtained during the storage period in both types of oils (ONPD and OOPD), the amount of Totox has increased. According to Figure 2, the highest amount of acidity (0.6) was observed for the OOPD in two time periods (120 and 150 days), whereas the lowest amount for the ONPD (0.4) was observed in the storage period of 90 days. These results were consistent with the results obtained by Ngando et al. (2011). The amount of peroxide on the first day for the ONPD and OOPD was 3.36 and 3, respectively. According to the result obtained for the peroxide index, the effect of parameter A (oil type) was not significant at the level of 1% probability. The peroxide index is a function of oxidation time and increases with the opening of the oil door and the onset of oxidative reactions. The peroxide index has an ascending trend and has reached its maximum 6.6 (meg O_2/kg) at the end of 150 days. According to Figure 2, the highest amount of peroxide (6.6) was observed in the ONPD on day 150, whereas the lowest amount of peroxide (3) was observed in the OOPD on the first day. Unlike the peroxide index, the anisidine shows the oxidation secondary products due to the degradation of hydroperoxides (Bonilla et al., 2012). According to Figure 2, the anisidine of the OOPD was higher than the ONPD. This index initially shows an increasing trend and then decreases slightly, which may be related to the complete destruction of hydroperoxides. The Totox index is an indicator for measuring total oxidation, that is, the primary and secondary products of oxidation (Shahidi & Zhong, 2021). The highest index of Totox (22.3) was observed in the OOPD after 150 days, whereas the lowest index of Totox (12.63) was observed on the first day for the ONPD.

3.2 | **Data exploratory analysis**

The voltage response of the sensors was measured in three replicated for 40 oil samples in a 150-day period at 1-month intervals. The mean of the sensor array response was then recorded for 240 samples. An 8×240 feature matrix was considered for input of the data analysis methods. Nonsupervised methods such as PCA are the first step if data analysis. The data obtained from the eight sensors were reduced two-dimensionally using PCA. This method is suitable for the data analysis with no prior knowledge on the samples class, where the objective is to form a hypothesis not confirming that. Two-dimensional score diagrams of the shelf life of the oil samples are depicted in Figure 3. The two main components managed to describe 97% of the data set variance (PC1 = 91% and PC2 = 6%) in discriminating the shelf life of the oil samples. As can be observed, nonoxidized oils are on the left side of the score diagram, whereas the spoiled ones were placed on the right side. It can also be seen

that the data for each month showed proper differences during the storage time. The results of the AOCS methods confirm the initial classification obtained in the score plot. Rasekh and Karami (2021a) reported similar results for predicting fruit fraud.

Accordingly, MQ136 and TGS2602 sensors possessed the highest significance in volatile pattern recognition and could be the best choice in the determination of the shelf life of the oil. On the other hand, TGS813, TGS2620, MQ9, and MQ135 had the lowest mean values and influence in sample discrimination.

3.3 | **ANN result**

Perceptron neural network was used for the classification of the shelf life of the oil. For this purpose, three different analyses were used to examine the shelf life (based on the storage time [150 days or 6 periods] for two types of oil [ONPD and OOPD]), differentiate the healthy oil from spoiled one, and investigate the healthy oils of both groups (ONPD and OOPD). For the input layer, according to the number of sensors, eight neurons were considered; and for the output layer, it was determined based on the type of the experiment (12, 2, and 6 neurons, respectively). Equations 7–11 were used to find the optimal neurons in the hidden layers. In this regard, the network with the structure of 8-15-12 exhibited the highest accuracy. The network training was conducted by logarithmic sigmoid transfer function and learning method of Lunburg–Markorat. RMSE and R² of the train and test data, as well as the correct classification rate of the best structure, are listed in Table 2.

According to the results, the network with the topology of 8- 15-12 exhibited the best performance with the lower train and test error and a higher coefficient of determination. To this end, the network with the topology of 8-10-6 possessed the best performance in differentiating the nonoxidized oils in a way that its RMSE was 0.001 and 0.01 for the training and test, respectively. Moreover, the mentioned topology resulted in R^2 values of .99 and .93 for the train and test data, respectively. In another experiment to differentiate nonoxidized from oxidized oils, the topology of 8-5-2 resulted in the best performance as it exhibited the highest accuracy for the train and test data. The obtained values were far higher than those reported by Ayari et al. (2018a) for the detection of the oxidation in animal and vegetable oils. These results had also higher accuracy compared with the work of Yu et al. (2008) concerning green tea classification using back propagation error ANN based on the data provided by an E-nose. Rasekh and Karami (2021b) also reported similar results for predicting fruit fraud.

The confusion matrix and classification performance parameters are shown in Figure 4 and Table 3. Among 240 data for the determination of the shelf life, discrimination of the healthy oils with two types of oil (ONPD and OOPD), and detection of the oxidized oils from nonoxidized ones, the proposed method managed to correctly allocate 224, 237, and 239 data in their corresponding groups. As Table 3 suggests, the ANN method offered **EXARAMI ET AL.** 9 of 10

high accuracy and sensitivity in the classification of the edible oils.

The results of this study are in agreement with the work of Wei et al. (2009) who used an E-tongue to classify honey samples with different flower and geographical origins. Their results were then analyzed by different pattern recognition techniques of PCA and ANN. They showed that ANN is the most effective feature extraction method in comparison with CA and PCA methods with a correction level of 95% (Wei et al., 2009). These results were also better than the other studies using ANN methods (Hai & Wang, 2006; Kiani et al., 2017; Singh et al., 2014).

4 | **CONCLUSION**

The oil durability studies were conducted under normal conditions for 150 days. These experiments were repeated each month using both the chemical method of AOCS and the proposed E-nose, which led to the following results:

- E-nose can be exploited for the shelf life and oxidation of the oil with satisfactory results.
- During the storage time of the oil samples, PCA and ANN methods succeeded in classifying the samples based on their storage time with high accuracy.
- The correlation between the measured and predicted smell parameters of the edible oil revealed high prediction performance based on the output signals of the E-nose.

Generally speaking, as a few studies have addressed the use of E-nose in the determination of the shelf life of oil, the results of this study indicate that E-nose, in combination with ANN, can be used to determine the shelf life of oil with satisfactory results.

CONFLICT OF INTEREST

The authors have declared no conflict of interest for this article.

AUTHOR CONTRIBUTIONS

Hamed Karami: conceptualization methodology, software, statistical analyses and data validation, formal analysis, investigation, writing original draft preparation, writing—review and editing. Mansour Rasekh: resources, statistical analyses, and funding acquisition. Esmaeil Mirzaee-Ghaleh: resources, review and editing, software, formal analysis.

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.

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