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Application of Electronic Nose and Eye Systems for Detection of Adulteration in Olive Oil based on Chemometrics and Optimization Approaches

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Abstract: In this study, a combined system of electronic nose (e-nose) and computer vision was developed for the detection of adulteration in extra virgin olive oil (EVOO). The canola oil was blended with the pure EVOO to provide adulterations at four levels of 5, 10, 15, and 20%. Data collection was carried out using an e-nose system containing 13 metal oxide gas sensors, and a computer vision system. Applying principal component analysis (PCA) on the e-nose-extracted features showed that 93% and 92% of total data variance was covered by the three first PCs generated from Maximum Sensor Response (MSR), Area Under Curve (AUC) features, respectively. Cluster analysis verified that the pure and impure EVOO samples can be categorized by e-nose properties. PCA-Quadratic Discriminant Analysis (PCA-QDA) classified the EVOOs with an accuracy of 100%. Multiple Linear Regression (MLR) was able to estimate the adulteration percentage with the R^2 of 0.8565 and RMSE of 2.7125 on the validation dataset. Moreover, factor analysis using Partial Least Square (PLS) introduced the MQ3 and TGS2620 sensors as the most important e-nose sensors for EVOO adulteration monitoring. Application of Response Surface Methodology (RSM) on RGB, HSV, L^*,a^*, and b^* as color parameters of the EVOO images revealed that the color parameters are at their optimal state in the case up to 0.1% of canola impurity, where the obtained desirability index was 97%. Results of this study demonstrated the high capability of e-nose and computer vision systems for accurate, fast and non-destructive detection of adulteration in EVOO and detection of food adulteration may be more reliable using these artificial senses.

Keywords: Fraud detection, Machine learning, Volatile Matter, Image Processing, Optimization.
Categories: J
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1 Introduction

Using vegetable oils in food production is a proper alternative to animal oils since the former contains fewer unsaturated fatty acids compared to the latter [Tahri et al., 2018]. Vegetable oils are fats extracted from plants produced in solid or liquid forms. The main use of these oils is in cooking and the industries related to the production of fuel, and health and beauty products. The most commonly used types of vegetable oils include olive, soybean, canola, and sunflower oils [Rizwan et al., 2017, Vidigal et al., 2021]. Providing enough food is one of the most important issues in the world concerning the increase in population rates, and providing healthy food to the community is just as important.

Food fraud is an old problem that is still an issue affecting the entire food industry, customers and consumers [Robson et al., 2021]. Based on the definition by the World Health Organization (WHO), adding any illegal and harmful substance to the human and animal food basket is considered fraud. Profiteers sometimes resort to food fraud to reduce production costs and make more profit, endangering human health [Hong et al., 2014, Teixeira et al., 2021].

Food fraud is divided into low-risk and dangerous frauds. Further, there are other categories in terms of nature, including selling one type of food instead of another, mixing one type of nutrition with another, not observing a registered formula or standard, supplying spoiled food, using non-edible colors and essence, and increasing or decreasing the components of a food [Du et al., 2018]. In addition to damaging food nutrition, oil fraud results in bad consequences for consumers, identifying food fraud is very important from health, economic, and social aspects [Sciuto et al., 2017]. Therefore, applying new methods with fast and high response levels is necessary to identify additives in oil [Hong and Wang, 2014]. One of the most common oil fraud cases includes adding cheaper vegetable oil to more expensive oil [Alvarez-Galvan et al., 2018, Tan et al., 2021].

Experimental analysis of oil provides comprehensive information about the content and composition elements of oil. High-performance liquid chromatography (HPLC) is one of the most routinely employed tools [Cordella et al., 2002]. The presence of sunflower oil in olive oil was investigated by Bakre et al. [2015] using reversed-phase HPLC based on α-tocopherol as the discriminating parameter. Gas chromatography (GC-MS) was used for adulteration detection in olive oil by separating volatile organic compounds [Jabeur et al., 2014].The use of DNA analysis has been used to test the authenticity of olive oil as a high-reliability tool [Kalaitzis and El-Zein, 2016]. There are good reviews on adulteration detection techniques in edible oils [Salah and Nofal, 2021], especially in extra virgin olive oil (EVOO) [Meenu et al., 2019]. However, the experimental techniques are mostly destructive, time-consuming, and tedious, and require expertise. Therefore, the development of non-destructive and accurate testing techniques for olive oil adulteration detection is still highly interested.

Electronic nose (e-nose) includes a number of semi-selective sensors and uses mathematical methods and signal processing based on pattern recognition or multivariate data analysis [John et al., 2021]. The output data can be analyzed by chemometric methods. Data processing algorithms such as principal component analysis (PCA) for classification and detection, and partial least squares (PLS) for calculating quantitative multivariate, along with other methods as a computational method for examining nonlinear sensor signals can be similar to the performance of the human brain [Ha et al., 2015, Al-Dayyeni et al., 2021]. Applications of electronic nose for the detection of food adulteration have been recently reviewed by Roy and Yadav [2021]. Teixeira et al [2021] studied application of electronic nose for olive oils commercial classification. E-nose-MOS allowed semi-quantitative and quantitative concentration assessment and discriminated oils with different ripe and green intensities. Cano Marchal et al [2021] studied fruity aroma intensity in virgin olive oil using an electronic nose. The results obtained were a mean validation error of units for the prediction of fruit aroma by regression with 88% accuracy for the defect detection using logistic regression.

In addition, image processing is one of the non-destructive methods of food monitoring. Computer vision is the technology used for automated inspection based on image processing. The computer vision system mainly consists of image acquisition devices and image processing software [Narendra and Hareesha, 2010]. An imaging device consists of a lighting system and a camera. After receiving the desired image, image processing algorithms are applied to the input image to extract the required information [Buratti et al., 2018]. This technique has been successfully used in assessing the quality of fruits, vegetables, and food in recent years [Kakani et al., 2020, Meenu et al., 2021].

Response Surface Methodology (RSM) is a set of solutions used to evaluate the optimal operating conditions through experimental methods, which involves performing various experiments, using the results of one experiment to guide the future path. Thus, the RSM is an optimization algorithm in which mathematical and statistical techniques are used [Kilic et al., 2019] to determine the optimal combination of the input factors that maximize or minimize a given objective function [Sagbas et al., 2021]. In general, RSM includes steps such as data coding, design, fitting the response model, and representing the response level graph [Askari et al., 2006, Mahmodi et al., 2022]. The concept and applications of RSM for optimization in the food industry are represented in a review article [Kidane, 2021] and a book chapter [Malekjani and Jafari, 2020].

In recent decades, applying fast, selective, and sensitive methods have been developed in food safety and health. Thus, industries are developing strategies with high responsiveness and low manufacturing costs. However, the use of traditional analytical techniques is limited by the need for expensive instrumentation and complex sample preparation. One of the most promising ways of developing rapid, sensitive, and inexpensive methods for quality control in virgin olive oils is the use of multi-sensor systems. Therefore, using electronic tools is necessary to evaluate foods with high accuracy and response. So, this study aimed to investigate the adulteration of EVOO with canola oil by the integration of computer vision and e-nose as non-destructive systems, combined with chemometric algorithms.

2 Materials and Method

2.1 Sample Preparation

In order to perform the experiments, EVOO was provided from Rudbar Olive Research Station, Guilan province, Iran. One of the most common adulterations of EVOO in Iran is adding canola oil into the pure EVOO. In this study, the edible canola oil was purchased from a market and was blended with pure extra olive oil to provide impurities at four levels of 5, 10, 15, and 20%. Pure and adulterated EVOO samples were stored at the refrigerator temperature of 4 °C until the experiments. Eight samples were examined with a net weight of 150 g for each class.

2.2 E-nose setup and procedures

The experiments were conducted in the laboratory of postharvest technology, Department of Biosystem Engineering of the University of Guilan. A laboratory-developed e-nose was employed for odor information acquisition. The system included an air capsule, a sensor chamber, a sampling chamber, data acquisition and collection system, and airflow valves. The schematic diagram of the olfactory machine system and the sensor array are demonstrated in Figure 1. The sensor chamber consisted of 13 metal oxide semiconductor (MOS) gas sensors, which are characterized by long life, extremely low response to moisture, high chemical stability and low price. Each sensor responds to specific combinations of volatiles in the chamber (Table 1). The gas sensors employed in this study were from TGS (Figaro Electronic Co., Ltd.) and MQ (Hanwei Electronics Co., Ltd.) families.

The operating procedure of the olfactory system includes baseline correction, measurement, and cleaning the sensor chamber. The baseline correction step was performed to stabilize the sensor array response. At this step, the pure oxygen was injected into the sensor chamber for 60 seconds. After that, a 40-second step was considered for the sample headspace odor acquisition phase in which the carrier gas transferred the gas from the oil headspace into the sensor chamber.

The last step was the cleaning phase, to bring the sensor response to the baseline and prepare the system for further tests. During this stage, the oxygen was injected into the sensor chamber for 60 seconds.

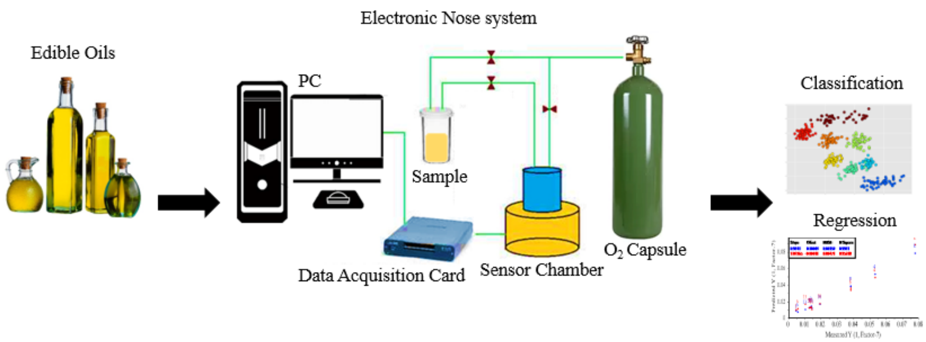


Figure 1: Schematic overview of e-nose system

No.	Sensor name	Detection ranges (ppm)	Main applications
1	MQ135	10-300 NH_3 10-1000	NH_3,NO_x, alcohol, Benzene, smoke,CO_2
2	MQ2	300-5000-LPG and propane	LPG, i-butane, propane, methane ,alcohol, Hydrogen, smoke
3	MQ7	100-300	CO
4	MQ9	20-2000 CO	CO and CH_4,LPG
5	MQ-3	0.05-10	Alcohol
6	TGS2620	50-5000	Alcohol, toluene, xylene, other volatile organic vapors
7	TGS813	500-10000	Domestic gas leak detectors and alarms,
8	TGS822	50-5000	Breath alcohol detectors, Gas leak detectors/alarm,
9	TGS2610	500-1000	Butane, liquid petroleum gas
10	TGS2602	1-30	Air cleaners, Ventilation control
11	MQ5	200-10000	LPG, natural gas, town gas
12	MQ8	100-1000	H_2
13	MQ4	300-10000	Natural gas/ Methane

Table 1: The specifications and applications of sensors used in the sensor array

2.3 E-nose Data Processing

Before extracting the e-nose-based features, pre-processing was applied to the recorded information. First, the size of the data was reduced during a feature extraction step. The type of analyte can be identified by classifying the data in the feature space [Nardecchia et al., 2020]. The fractional manipulation (Equation 1) was employed on the raw data to provide a dimensionless and normalized response. In equation 1, the ys(t), xs(0), and xs(t) stand for the preprocessed response, the baseline value in the raw response, and the dynamic raw response of the sensors, respectively [Hai and Wang, 2006, Heidarbeigi et al., 2015]. In this study, the baseline manipulated responses of sensors during the headspace odor acquisition stage were used for feature extraction (Figure 2).

$$y_s(t) = \frac{x_s(t) - x_s(0)}{x_s(0)} \quad (1)$$

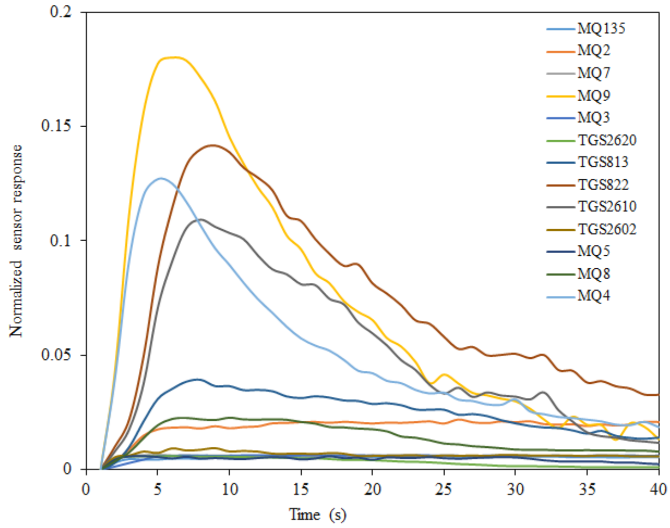


Figure 2: A sample pre-processed response of sensor array

After preprocessing, a combination of transient and steady-state information from the sensor responses was used for adulteration detection. It was reported by Nimsuk [2019] that the integration of both types of features can improve the e-nose performance. In order to provide the e-nose data vector, six different parameters were extracted from the manipulated signal curve of each sensor. These features were the Maximum Sensor Response (MSR), the response Area Under Curve (AUC) during the headspace acquisition stage, the time to reach the MSR (TR); the average slope of the rising portion of the sensor response (Sasce), the maximum instantaneous slope (Smax); and the Mean Differential Coefficient Value (MDCV) which was the average velocity of sensors responses. The mentioned features have been described and used in previous literature [Xu et al., 2017, Nimsuk, 2019, Marek et al., 2020]. The Extracted e-nose data are available as supplementary to this article.

2.4 Image acquisition and processing

The original top-view images of the oil samples were acquired using an image acquisition system consisting of a digital CCD camera (Samsung ES95 digital color camera, Korea, 10 MP), and a wooden illumination chamber equipped with 12 VDC LED light strips as the lighting system. The camera was placed at a vertical distance of 0.2 m above the oil samples which were on matte white cardboard. The size of the captured images was 3648×2736 pixels. The color images were transferred into the laptop computer and read in the image processing toolbox of MATLAB programming software (Version 2021a, the MathWorks, USA) for processing and feature extraction operations. First, a 1200×1200 block around the center of the image was cropped containing the oil sample (Figure 3a) to remove some of the invalid pixels at the image edge regions. In order to segment the oil regions from the image background, the

Excessive Red (EXR) component (Figure 3b) of the cropped RGB image was calculated using equation 2.

$$EXR = 2 \times R - G - B \tag{2}$$

where R, G, and B are the Red, Green, and Blue color components of the cropped RGB image, respectively. By optimal thresholding on the EXR image, the primary binary image of the oil was obtained. After removing the boundary regions of the segmented image, and filling probable holes inside the region of interest, the final binary image of the oil was resulted (Figure 3c). Finally, by applying logical AND operation between the binary image and color image, the RGB image of oil was obtained with zero value in the background regions (Figure 3d). This image was then transformed from the RGB color space to HSV (Figure 3e) and L*a*b (Figure 3f) color spaces, and the average values of red, green, blue, hue, saturation, value, lightness, and a* and b* color components were extracted. The Extracted color data are available as supplementary to this article.

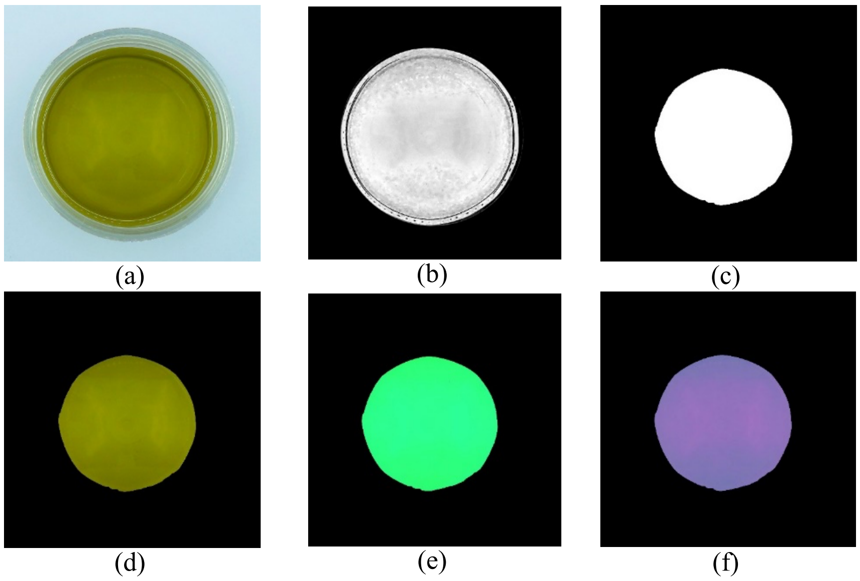


*Figure 3: Gallery of the images resulted from different steps of image processing, a) cropped RGB image, b) EXR image, c) binary image after removing boundary regions and filling the holes in region of interest (ROI), d) RGB image of oil with zero for background regions, e) HSV image of oil with zero for background regions, and f) L*a*b image of oil with zero for background regions*

2.5 Data Analysis Methods

2.5.1 Cluster analysis

Cluster analysis is an unsupervised pattern recognition algorithm that categorizes unlabeled items into clusters that are more similar together than the data in other clusters [Sharma, 2021]. The closeness of a sample to a class is measured by a specific distance metric [Sarno and Wijaya, 2019]. The clustering algorithm with the classical Wards method, and the squared Euclidean distance as the dissimilarity measure [Karami et al., 2020], was applied to the different types of extracted data to investigate the capability of odor characteristics for differentiating different levels of adulteration in EVOO. The Wards method clusters the data into groups that have the lowest interior variance [Muthahharah and Juhari, 2021].

2.5.2 Principal Component Analysis (PCA)

PCA involves the use of new functions to examine patterns. By applying PCA, the initial variables are transformed into new and independent components with a zero correlation coefficient for both main components. The newly created components are a linear combination of the initial variables [Amirvaresi et al., 2021]. In other words, PCA is a linear conversion taking data to a new coordinate system so that the largest variance of the data is on the first coordinate axis, the second largest variance is on the second coordinate axis, and so on. PCA can be used to reduce the size of the data, thus preserving the components of the data set with the greatest impact on variance [Keshavarzi et al., 2020]. In this study, PCA with cross-validation was applied to the different non-destructively obtained data, and the resulting score matrices were compared to find the most representative components to classify pure and adulterated olive oils.

2.5.3 Discriminant Analysis (DA)

DA is one of the most widespread multivariate analysis techniques that has been applied to differentiate objects into pre-defined groups [Siqueira et al., 2018]. The technique is detailedly described in a book authored by [McLachlan, 2005].
In this study, the principal components (PCs) generated from the e-nose extracted features were fed into two common Discriminant Analysis (DA) algorithms of Linear DA (LDA) and Quadratic DA (QDA) with cross-validation strategy for categorization of pure and adulterated EVOOs. DA algorithms with different numbers of PCs (1 PC to 8 PCs) were compared to find the most accurate discriminant classifier.

2.5.4 Discriminant Analysis (DA)

MLR is a supervised learning algorithm that is defined as the extension of simple linear regression when there are two or more explanatory variables [Lee et al., 2022]. MLR achieves the best fit by minimizing the sum of squares of differences between the actual and predicted values [Siedliska et al., 2018]. The MLR algorithm with leverage correction validation method was used in this research to find the possible relationships between the sensory extracted information and the adulteration percentage, and therefore to predict the adulteration rate based on odor characteristics.

2.5.5 Partial Least Square (PLS)

One of the most powerful techniques in the field of chemometrics is factor analysis which is employing a multivariate algorithm to reduce the number of input variables from one $i \times j$ input matrix to a combination of factor j [Mohammad-Razdari et al., 2019]. PLS can efficiently resolve multiple data, correlation, and overlap problems, and has a high ability to identify the best sensors among different sensors having intercorrelations [Rasekh et al., 2021]. In this study, PLS with cross-validation was applied to the e-nose extracted data to find the most appropriate sensor(s) for differentiating between different EVOO samples based on odor emission. Based on the minimum value for RMSE and the maximum value for R^2, the most important sensors were selected.

2.5.6 Optimization of color parameters with RSM

The effect of the oil type as the independent variable on the color parameters as dependent variables was investigated using the optimal custom design of RSM. This optimization method includes a number of design points and a repeating center point. It was assumed that there are three mathematical functions (f_k) exist for y_k as follows:

$$y_k = f_k(x) \tag{3}$$

where x is the type of oil, which is the natural variable based on its natural units. In RSM, the natural variable was transformed into a dimensionless coded variable (X) with a zero mean. In the present study, a second-order polynomial model was used to associate the responses to the factor [Yesilyurt et al., 2019, Zhang et al., 2019]:

$$y_k = \beta_0 + \beta_1 X + \beta_2 X^2 + \varepsilon \tag{4}$$

where y_k is the predicted response which was considered as a dependent variable (k=1, 2,…, 5), the β coefficients stand for regression coefficients where β_0 represents the offset term, and β_1 and β_2 represent linear effects, and ε is the random error term. The value of the independent variable was coded between -2 and +2 (Table 2). In this design, 9 experimental runs were augmented with five replications at the center point to evaluate the pure error (Table 2).

Independent factors	**Coded factors**	**Natural factors**	**Levels**				
Oil Classes	X	x	-2	-1	0	+1	+2

Table 2: Independent test variables and selected levels in the RSM analysis

The cluster analysis, PCA, DA, MLR and, PLS analyzes were performed using Unscrambler x10.4 software (CAMO AS, Trondheim, Norway), and RSM analysis was carried out using Design-Expert.10.0.6.0 (Stat Ease Inc, USA).

3 Results and Discussion

3.1 Results of cluster analysis

The results of clustering of pure and impure EVOO samples by the Wards method using the squared Euclidean method are depicted in Figure 4. As shown in this figure, the MSR, AUC, Smax, and MDCV properties have a high capability to classify pure and impure samples. In addition, although there are some misjudgments in the case of TR and Sasce traits, however, the high potential of the used clustering method with most of the e-nose data is promising so that the odor-extracted features can be used for differentiating the pure and adulterated EVOOs with high accuracy. The applicability of the Wards clustering method was also reported by Karami et al. [2020] for the detection of adulteration in fresh edible oils by oxidized, where the classes contained fresh oil (no adulteration), 25% adulteration, 50% adulteration, and fully oxidized oil (100% adulteration).

3.2 PCA results

Based on PCA results, a significant difference was observed between pure and adulterated olive oil samples. Figure 5 exhibits that the first three PCs of most of the extracted properties were able to describe the high variance of the data. It was observed that the MSR, AUC, TR, Sasce, Smax, and MDCV features could identify 92% (PC1 = 50%, PC2 = 37%, PC3 = 5%), 93% (PC1 = 55%, PC2 = 32%, PC3 = 6%), 67% (PC1 = 28%, PC2 = 26%, PC3 = 13%), 82% (PC1 = 47%, PC2 = 22%, PC3 = 13%), 88% (PC1 = 55%, PC2 = 27%, PC3 = 6%), and 88% (PC1 = 48%, PC2 = 31%, PC3 = 9%) of the total data variance, respectively.

The presented view in Figure 5a shows that the MSR feature was able to differentiate the pure EVOO from the adulterated samples. It is also observed that the AUC (Figure 5b), TR (Figure 5c), Smax (Figure 5e) and MDCV (Figure 5f) features could identify the pure EVOO from adulterated samples using the first three components with appropriate accuracy. These results indicate that there is a high capability to distinguish pure EVOO from impure ones using appropriate odor-extracted features.

3.3 Adulteration detection using PCA-DA

Several LAD and QDA models, with different numbers (1 to 8) of PCs as input data, were developed and compared to select the best adulteration classifier. Results of the evaluated DA algorithms are provided in Table 3. Results showed that, in most cases, the PCA-QDA models were more accurate than the PCA-LDA algorithms with the same number of input PCs. Furthermore, the classification ratio increased by increasing the number of PCs as the input parameters. The highest classification ratio of 100% was obtained by PCA-QDA with different numbers of PCs generated from AUC, TR, Sasce, Smax, and MDCV features as the input parameter. This shows the high potential of e-nose features for the separation of all the pure and adulterated EVOO samples. Among the models with 100% accuracy, the AUC-PCA-QDA and TR-PCA-QDA structures with five input PCs (AUC-PCA5-QDA and TR-PCA5-QDA classifiers) were selected as the most appropriate classifiers due to their minimum number of input PCs to reach the highest performance. Graphical visualizations of these two classifiers are available in Figure 6 in the plane of second vs. first discriminant functions. These models were able to classify the test dataset of pure and adulterated ones with an accuracy of 95%. Roy et al. [2021] reported that the DA method was able to discriminate the pure ghee (clarified butter fat) samples from the adulterated samples by 10% to 60% of commercial refined soybean oil. Their obtained accuracy was 94.54% on calibration data and 87.50% on test samples. Successful application of DA was also reported by Jiang et al. [2021] for shelf life classification of edible oils by e-nose made with four metal oxide semiconductor gas sensors, including TGS2600, TGS2611, TGS2620, and MQ138.

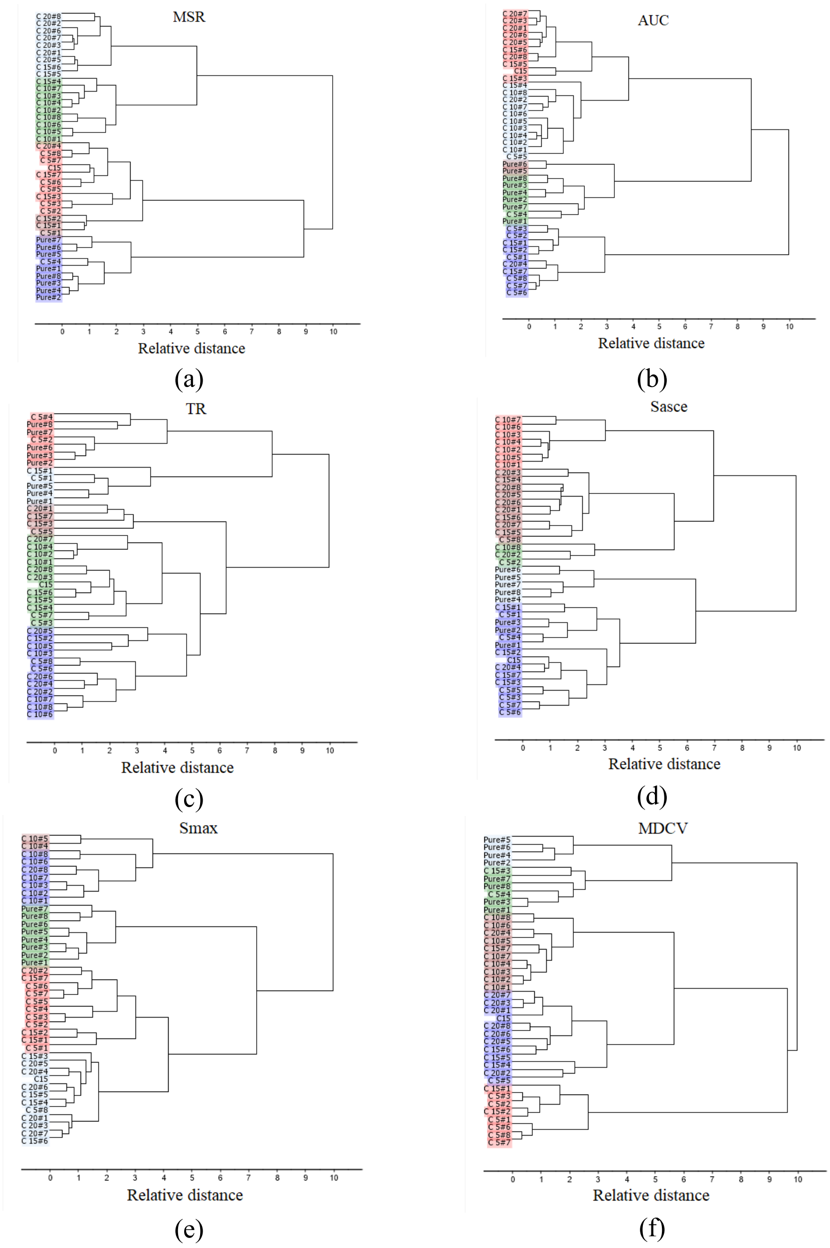


Figure 4: Results of detection and classification of the pure EVOO from the adulterated sample by KNN combining the characteristics of (a) MSR, (b) AUC, (c) TR, (d) Sasce, (e) Smax, , and (f) MDCV

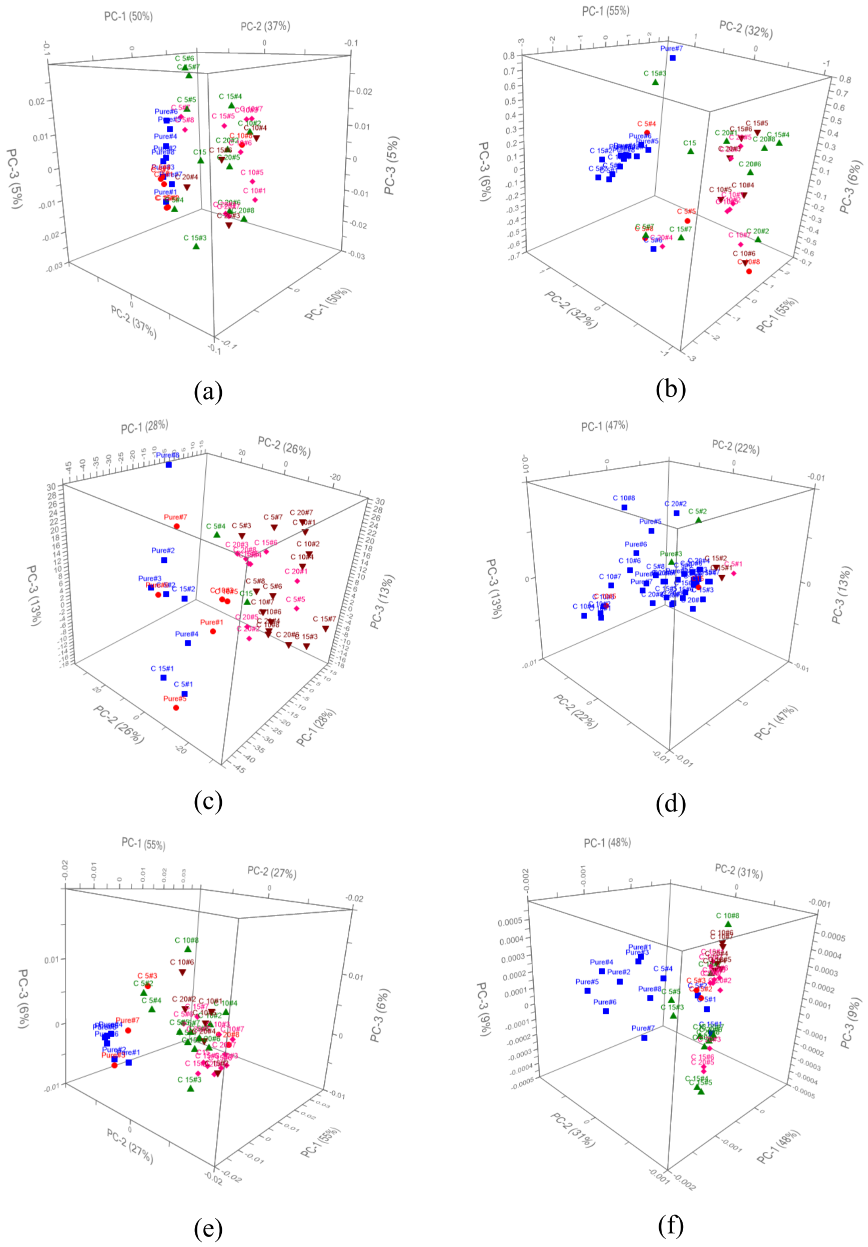


Figure 5: PCA 3d plots in the plane for classification of pure EVOO from adulterated samples, combining the characteristics of (a) MSR, (b) AUC, (c) TR, (d) Sasce, (e) Smax, and (f) MDCV

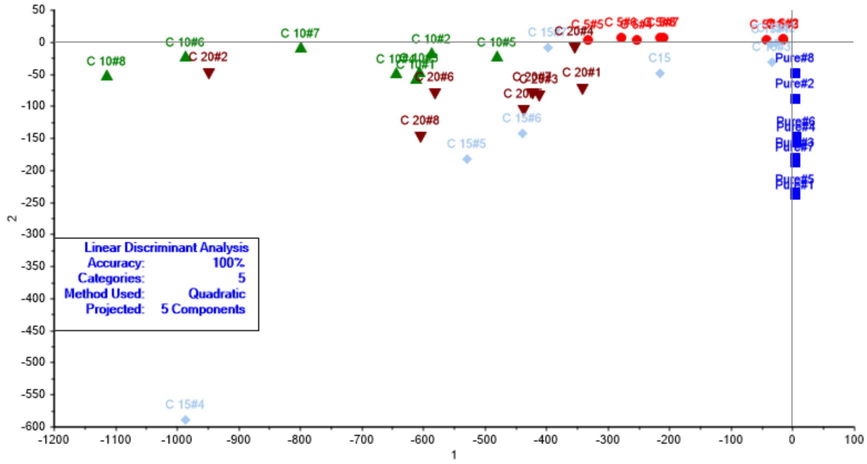
Classifier	**Number of PCs**							
	1	2	3	4	5	6	7	8
MSR-PCA-LDA	55	77.5	85	92.5	92.5	92.5	92.5	92.5
MSR-PCA-QDA	57.5	80	92.5	97.5	97.5	97.5	97.5	97.5
AUC-PCA-LDA	60	72.5	75	77.5	85	87.5	90	90
AUC-PCA-QDA	55	72.5	87.5	92.5	**100**	**100**	**100**	**100**
TR-PCA-LDA	35	47.5	50	85	85	92.5	95	95
TR-PCA-QDA	42.5	60	57.5	95	**100**	**100**	**100**	**100**
Sasce-PCA-LDA	52.5	77.5	87.5	87.5	85	90	90	87.5
Sasce-PCA-QDA	52.5	80	82.5	92.5	92.5	97.5	**100**	**100**
Smax-PCA-LDA	52.5	82.5	87.5	87.5	85	90	95	95
Smax-PCA-QDA	52.5	82.5	95	90	92.5	**100**	**100**	**100**
MDCV-PCA-LDA	52.5	62.5	72.5	82.5	82.5	90	87.5	90
MDCV-PCA-QDA	52.5	77.5	85	90	95	**100**	**100**	**100**

Table 3: Classification accuracy values (in percent) of DA structures for differentiation of pure and adulterated EVOOs

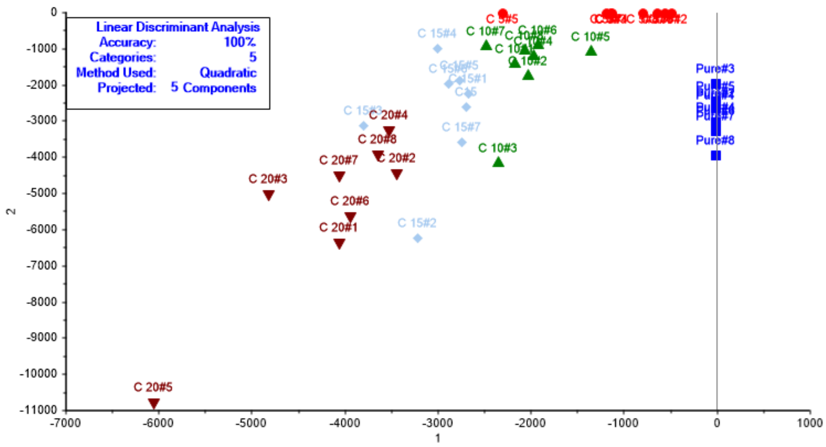
Another point from Table 3 is that the use of features such as AUC, TR, Sasce, Smax, and MDCV can increase the pattern recognition accuracy compared to classical MSR, which is usually used in e-nose research. Moreover, it should be mentioned considering Fig 5 and Table 3 that although the first three PCs of the TR feature covered a smaller amount of the total data variance when compared to other features, however, by increasing the number of input Pcs to 4 and five, the accuracy of TR-based DA increased. This shows that there was significant information in the PC4 and PC5 of the TR property for adulteration classification.

3.4 MLR results

Table 4 presents the performance values of MLR models for predicting the percentage of adulteration based on different extracted e-nose features. It was resulted that the MLR model based on TR property obtained the highest performance on both calibration and validation datasets. At the calibration stage, the R^2 and RMSE criteria of the TR-MLR model were 0.9505 and 1.9517, respectively. This model gave an R^2 of 0.8565, and an RMSE of 2.7125 on the validation dataset. Calibration and prediction results of the TR-PLS model for estimation of the adulteration rate of EVOO are graphically illustrated in Figure 7. Note that providing more experimental data with adulteration percentages closer to each other (e.g. 2%, 4%, 6%, etc.) may help to create a more accurate model and is suggested to be investigated.



(a)



(b)

Figure 6: Graphical output of the most accurate AUC-PCA5-QDA (a), and TR-PCA5-QDA (b) classifiers in the plane of second discriminant functions vs. first discriminant function for classification of pure and adulterated EVOOs (pure ■, 5% adulteration ■, 10% adulteration ■, 15% adulteration ■, 20% adulteration ■)

	calibration		Test	
Input feature	R^2	RMSE	R^2	RMSE
MSR	0.9230	2.4338	0.7961	3.2336
AUC	0.9079	2.6624	0.7408	3.6456
TR	**0.9505**	**1.9517**	**0.8565**	**2.7125**
Sasce	0.8792	3.0482	0.7153	3.8213
Smax	0.8031	3.8921	0.4574	5.2749
MDCV	0.8975	2.8073	0.7187	3.7982

Table 4: Performance criteria of MLR models for prediction of adulteration rate based on e-nose features

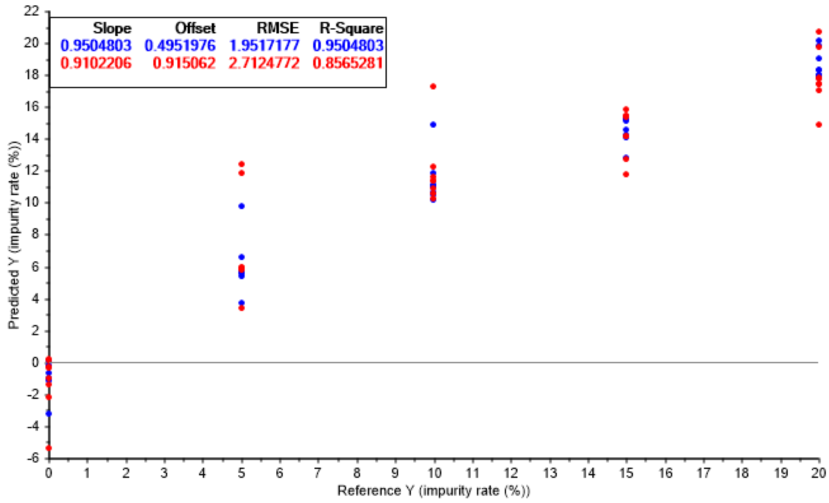
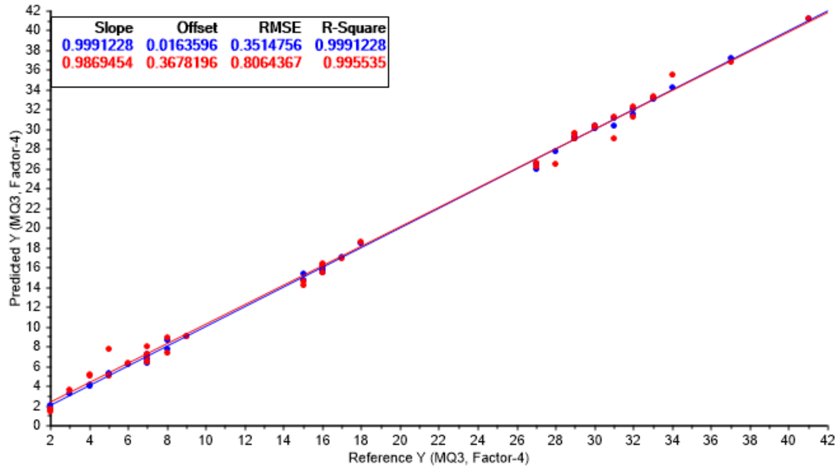


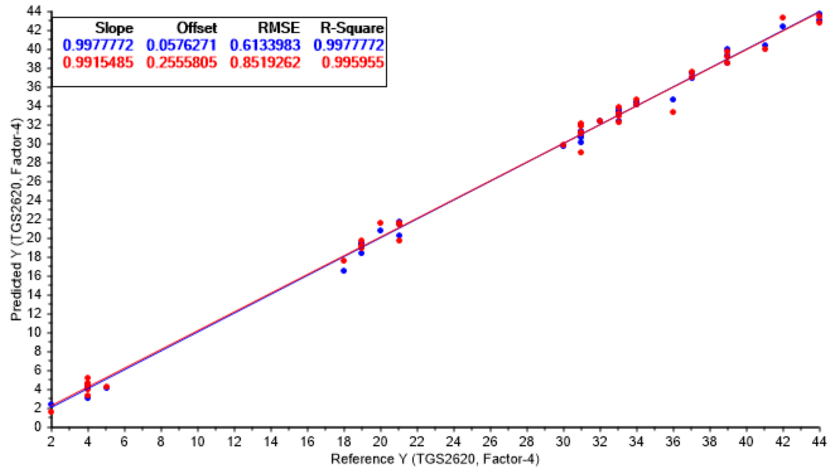
Figure 7: Graphical result of MLR model for estimation of impurity rate of EVOO based e-nose TR property (TR-MLR model) at calibration (blue) and prediction (red) stages

3.5 PLS results

Regarding that, the TR feature resulted in the most accurate classification and regression models when fed into QDA and MLR algorithms, the PLS was applied to the TR features of the sensor array to find the best sensors. Regarding the obtained R^2 and RMSE criteria at the validation stage, the TR measures of the MQ3 and TGS2620 sensors are the most important sensor-based features for EVOO adulteration monitoring. The high correlation between the PLS-predicted and the actual TR values of these sensors is illustrated in Figure 8. It was reported by Mohammad-Razdari et al. [2019] that the TGS2610 and MQ3 sensors are the most important sensors for authentication assessment in tomato paste. In another study, Rasekh et al. [2021] introduced the TGS813 and MQ135 sensors as the best sensors for discrimination of herb and fruit essential oils. Moreover, Karami et al. [2020] reported that the MQ136 and TGS2602 were the most significant sensors for monitoring edible oil oxidation based on the PLS method.



(a)



(b)

Figure 8: Linear correlation of predicted vs. actual (reference) TR values of the most significant individual sensors, a) MQ3 sensor, b) TGS2620 sensor

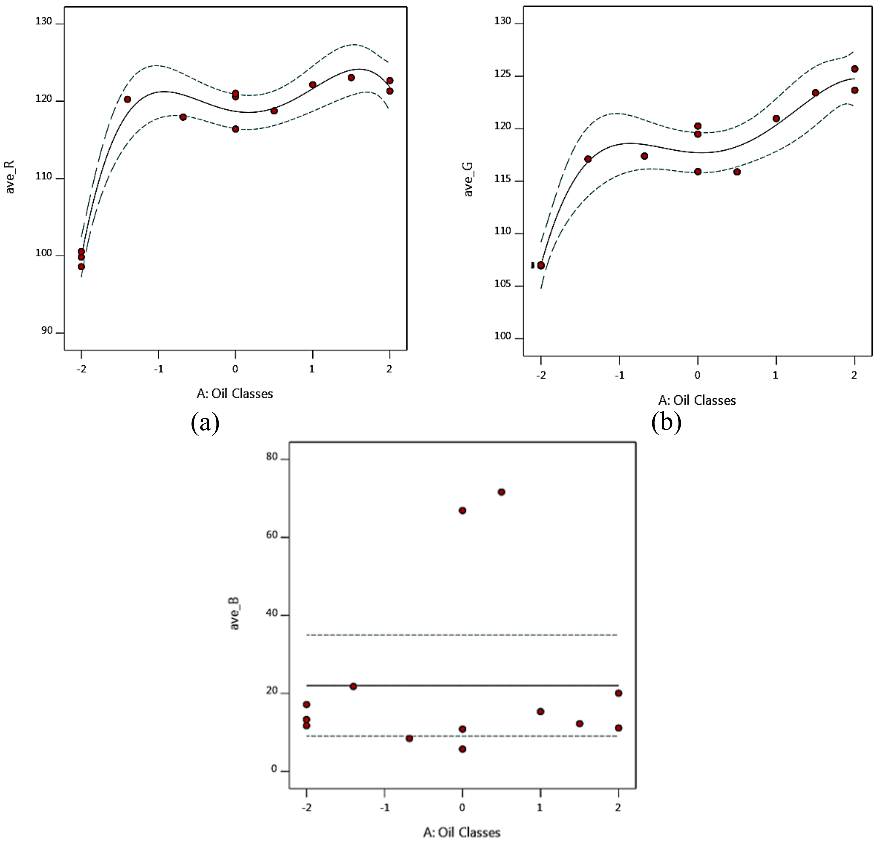
Product	Type of e-nose	Detection result to recommend best technique	References
Olive oil	Experimental type with 13 MOS gas sensors	PCA-LDA with 100% classification accuracy, MLR with R^2 of 0.8565 for prediction of adulteration rate	This study
Olive oil	Commercial type	PCA with 81% accuracy and LDA with 92% accuracy	[Teixeira et al., 2021]
Olive oil	Commercial type with 8 sensors	88% accuracy for PCA and 79% accuracy for LDA	[Cano Marchal et al., 2021]
Olive oil	Commercial type, Fox 4000 Alpha MOS e-nose with 3 sensor chamber each containing 6 gas sensors	PLS: correlation coefficient of 0.989 for rapeseed adulteration of olive oil while, 0.990 for sunflower adulteration of olive oil	[Mildner-Szkudlarz and Jeleń, 2010]
Peony seed oil	Commercial type, PEN3 e-nose with 10 MOS chemical gas sensors	LDA: soybean oil and sunflower oil can be successfully discriminated from peony seed oil	[Wei et al., 2018]
Soybean oil	Experimental type, 8 MOS gas sensors	PLS: correlation coefficient of 0.843 for old frying oil adulteration of soybean oil	[Men et al., 2014]
Argan oil Sunflower oil	Experimental type, e-nose system with 5 MOS gas sensors	PCA: 98.81% in comestible argan oil adulteration and 98.09% in cosmetic argan oil adulteration	[Bougrini et al., 2014]
Virgin coconut oil	Commercial type, Sensor Technology	PLS: correlation coefficient of 0.91 for palm olein oil adulteration of virgin coconut oil	[Marina et al., 2010]

Table 5: Comparison of the other studies results for adulteration in oils with this study

3.6 Optimization of Color Parameters of EVOO

Figure 9 to Figure 11 shows the results of optimizing the color parameters of EVOO samples. The changes in color parameters are presented against five classes of pure and adulterated EVOO including canola oil fraud of 5, 10, 15, and 20% with codes of C5, C10, C15, and C20, respectively (Figure 9 to Figure 11). The value of the red (R) color component in pure EVOO was low and increased by adding 5% of impurities. Further, by adding impurities and reaching to C10 sample, the value of R color component decreases slightly. Furthermore, the changes in the green (G) color component of the pure EVOO was considerably low and increased by increasing impurities. Based on the results, the amount of impurity does not affect the blue (B) color component.

The changes in the HSV parameters are depicted in Figure 10. The hue (H) value of the pure oil samples was high and reduced by adding impurities to the pure oil sample, which was more evident in C5 and C15 oils. Further, the saturation (S) parameter in the pure samples was low and increased by adding canola oil. Furthermore, the value (V) component was low in the pure oil, while it increased by adding adulterations.



(c)

Figure 9: Results of RGB color parameters in pure and adulterated EVOO samples, a) red, b) green, c) blue

The lightness (L*) component of the pure EVOO samples increased by adding impurities. The increase of lightness in the EVOO due to adding canola impurity may be because the crystals of the blended canola increased the light reflectance (Figure 11). In addition, the amount of a* parameter of pure EVOO samples was low, while it increased with the addition of impurities. There was no specific trend in a* parameter by increasing the adulteration level. Finally, the b* color chrominance was low in the pure samples and increased by increasing the level of adulteration. The a∗ represents green to red chromaticity and the b∗ represents blue to yellow chromaticity [Chavoshizadeh et al., 2020]. This shows that, regarding Figure 11a and Figure 11c, the EVOO appearance becomes brighter and yellower by adding canola impurity.

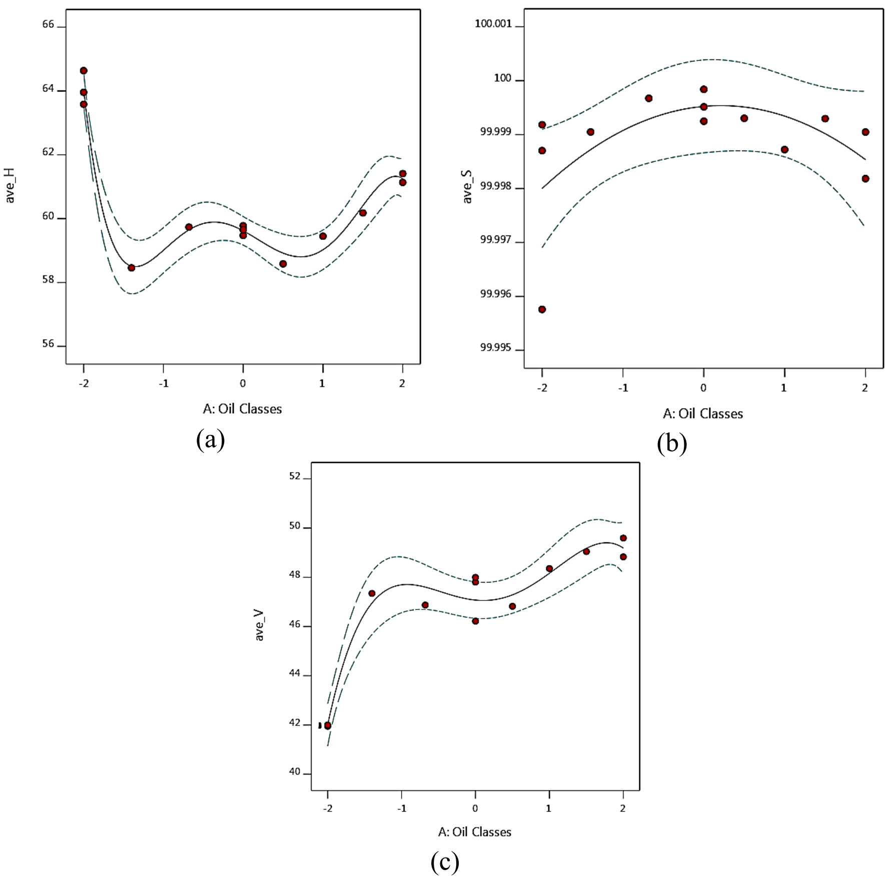


Figure 10: Results of HSV color parameters in pure and adulterated EVOO samples, a) hue, b) saturation, c) value

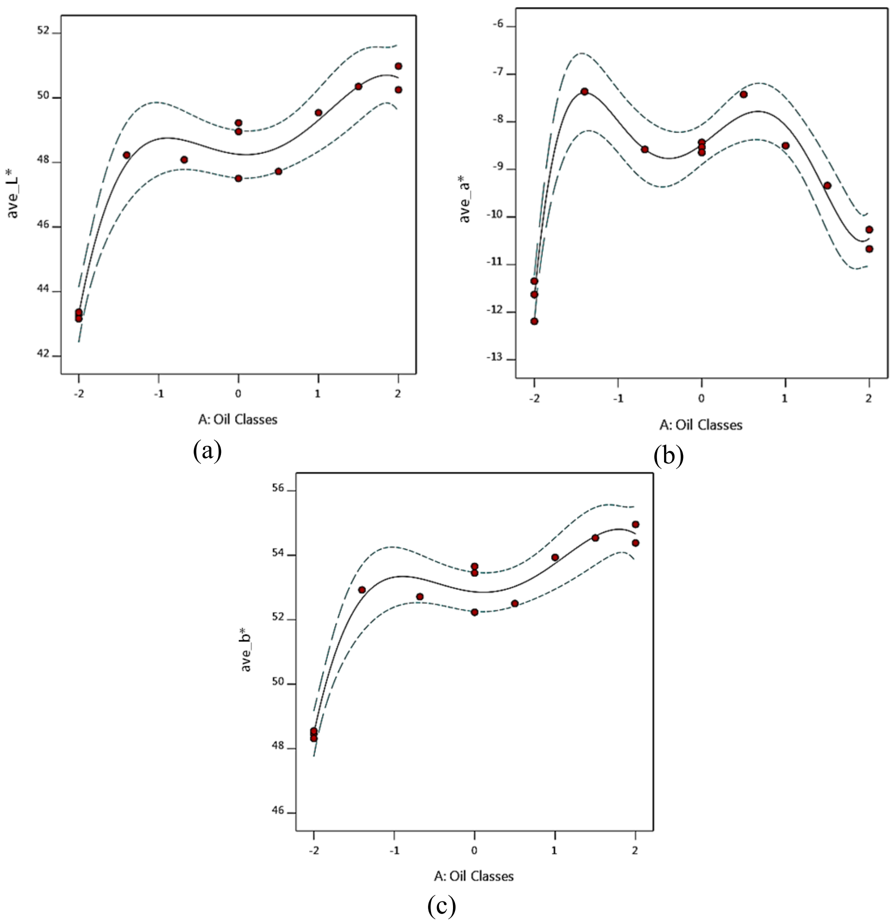


*Figure 11: Results of L*a*b color parameters in pure and adulterated EVOO samples, a) lightness (L*), b) a* chrominance, c) b* chrominance*

Figure 12 displays the results of the desirability ramp for numerical optimization of color parameters concerning the oil adulteration level. The desired targets for the optimization were selected from the information in Figure 9 to Figure 11 for pure EVOO. The results indicated that the color parameters were at their best and most optimal state in the case that the impurity level in EVOO was 0.099%. This means that the EVOO sample has its best values of color up to the adulteration rate of 0.099%. In other words, the addition of canola impurity into pure EVOO up to about 0.1% cannot be detected using image information. Furthermore, the desirability index in this modeling was 97%. Such high desirability value indicates the high accuracy of the model. Peris and Escuder-Gilabert [2016] reported the high effectiveness of combined e-nose and vision systems to detect the fraud and quality of edible oils.

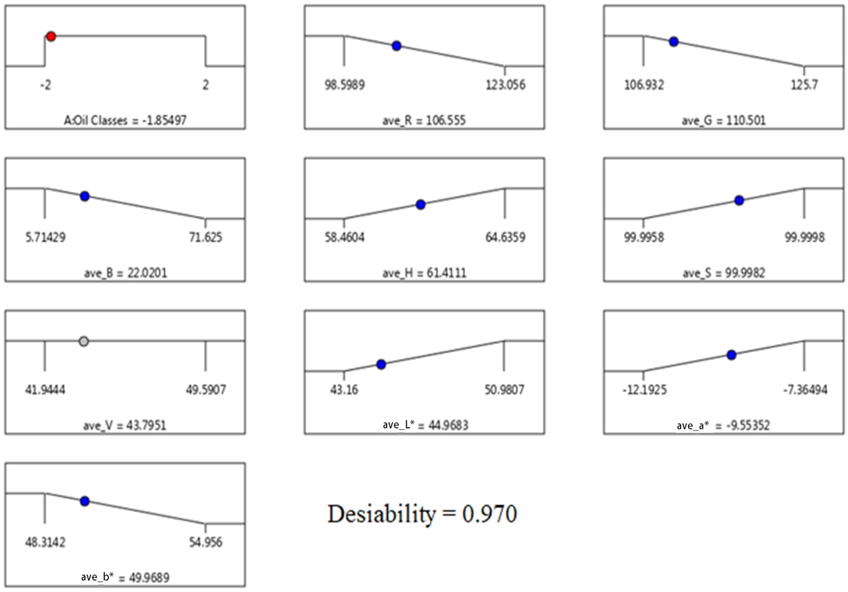


Figure 12: Desirability ramp for numerical optimization of EVOO color parameters by the RSM

4 Conclusions

Edible oils are widely used in various sectors. Therefore, the oil's health and safety are very important. So, studying new, accurate, and non-destructive methods for oil quality monitoring is highly interesting. In the present study, a combination of non-destructive methods of olfactory and vision machines was used to identify and classify pure EVOO from adulterated EVOOs with 13 sensors in combination with eye monitoring. The e-nose system in arrangement with clustering, PCA, DA, MLR, and PLS chemometric methods depicted considerable results. PCA and cluster analysis methods were able to categorize the data of 13 e-nose sensors with high accuracy. The QDA model was able to classify all the pure and adulterated EVOO samples by using the first five PCs generated from the TR feature of the e-nose system. The application of MLR for estimating the adulteration percentage resulted in high R^2 values in model calibration and validation stages. It was suggested to conduct experiments on the lower and closer rates of adulteration to obtain a more accurate and reliable estimator model. Also, PLS results showed that the MQ3 and TGS2620 sensors were the most important sensors for the classification of pure and impure EVOOs. Finally, the image-extracted color parameters were analyzed by RSM and the results revealed that the impurities in EVOO can be accurately identified by e-nose systems. The results of this study proved the high potential of e-nose and computer vision as non-destructive and non-contact systems for adulteration monitoring in EVOO that is fast and non-destructive other than methods. The use of both types of e-devices in this field has been steadily increasing along the recent decade, mainly due to the fact that their efficiency has been significantly improved as important developments are taking place in the area of data handling and multivariate data analysis methods. Finally, the use of electronic nose and eye is found in more food industries every day. Its technology continues to evolve; the future of sensory analysis will undoubtedly apply these mechanisms due to the reliability, speed, and reproducibility of the results.

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