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Research Article

A Novel Chemometric Learning Of Virgin And Deep Frying Olive-Oil By Fourier Transform Infrared Spectroscopy (FTIR)

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ARTICLE INFO

Article history:

Received 20 December 2023
Received in revised form 9 April 2024
Accepted 7 May 2024
Available online 30 June 2024

Keywords:

FT-IR, Chemometrik Method, Olive Oil, Deep Frying Oil.

Doi: 10.24012/dumf.1407248

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ABSTRACT

Olive oil is a very valuable product with its unique flavor, taste, aroma and bioactive components. It is an oil obtained by squeezing the olive with its pit and can be consumed in its natural form without any chemical treatment. It is well valuable thanks to its features such as helping healthy nutrition, contributing to economic and agricultural areas and being environmentally friendly. It is one of the most adulterated products today. So oil quality and content have to be determined properly. The aim of this study is to examine the machine learning of chemometrically fried oils in virgin olive oil and eight times used olive oil compared using Fourier Transform Infrared spectroscopy. Deep-Frying Oils (DFO) was carried out 8 times for 20 minutes. Because of chemical quality of oils, Cis, Trans, Ester, Methyl, Carbonyl, peroxide, unsaturated peroxide and ether groups were used in These results were evaluated by classification and regression using machine learning methods. For these evaluations, firstly classification and regression were made using all properties of these index. In classification models, Linear Discriminant Analysis, Naïve Bayesian and Ensemble Tree were used. The evaluation was carried out in two stages. In the first stage, half of the dataset was used for training and the other half for testing. In the second stage, all data was used for training and testing using cross validation method. The success results obtained using the all data set was 100% with naïve Bayesian method. According to chemometric strategy, differences between virgin olive oils and DFO were found by high accuracy in this study. This phenomenon also could be possible for other oil types and degrees of purity. Results illustrated that the method is very suitable and exact for detection deteriotion of olive oil.

Introduction

Olive oils (OO) are the product prepared from the olive of cultivated from olive trees (Olea europaea L.) Consumption of OO, which is the product of Olea europaea L., dates back to antiquity [1]. It has internationally accepted characteristic smell and taste [2]. On the other hand, OO is high cost product compared to other vegetable oils (sunflower oils, soy oils, corn oils, nut oils, peanut oils, hazelnut oils etc) [3].

Chemometry is defined as a multivariate data analysis method. It is used together with data-rich instrumental methods such as vibrational spectroscopic methods. These methods used to detect food trick and used for reducing data size, grouping samples qualitatively or classifying unknown samples and quantitatively detecting adulteration in sample [4]. Thanks to chemometric methods, scientific research will reach more accurate and faster results.

On the basis of machine learning (ML) algorithms, artificial intelligence applications are used efficiently in many sectors [5]. When the studies on the subject are examined; Bellou et al. used ML methods in OO classification in their study [6]. Stefas et al. used ML methods to determine the quality of extra virgin olive oil (EVOO) in different olive varieties obtained by Laser Induced Breakdown Spectroscopy (LIBS) and absorption spectroscopy [7]. Gyftokostas et al. aimed to classify 36 OO varieties obtained using LIBS from different parts of Crete, Greece, according to their geographical origin by using ML algorithms [8]. Yakar and Karadağ aimed to use different learning algorithms to identify adulterated OO in which olive oil is blended with different vegetable oils [9]. Drakopoulou et al. conducted a comparative study to evaluate the quality and authentication of Greek olive oils using ML methods [10]. Gonzalez Viejo and Fuentes used ML methods to evaluate the level of odor and aroma in commercial extra virgin olive oils by examining four different varieties using near infrared spectroscopy (NIR) and an e-nose [11]. Venturiini et al. used fluorescence spectroscopy to evaluate three different grades of olive oils using different ML methods [12]. Hou et al. investigated the feasibility of rapidly identifying adulterated EVOO using low-field nuclear magnetic resonance (LF-NMR) relaxation

measurement and ML approaches [13]. Gazeli et al. used ML methods to extract the information contained in the LIBS spectrum to classify olive oils according to their acidity [14]. Zhao et al. generated Raman spectra for ten commercial edible oils from various brands and analyzed them with supervised ML algorithms and then compared them with a principal component analysis (PCA) model [15]. Skiada et al. aimed to develop a classification model capable of olive variety identification based on olive oil chemistry [16]. Vega-marquez et al. combined chemical techniques and Deep Learning approaches to automatically classify three groups of olive oils corresponding to olive oil samples from two different harvests [17]. Gumus et al. evaluated different classification algorithms to reveal the most accurate one for authentication of Turkish olive oil [18].

The aim of the present work is to develop a novel application of the chemometric methods thanks to rapid, cheaper and nondestructive authenticity measuring tool, beneficial to determine the adulteration of virgin olive oils and 8 times deep frying olive oil by Transform Infrared spectroscopy (FTIR). This approach could be potential represents a quickly and more correct method.

Materials and Methods

Datasets

DFO data was used from research of Karaogul et al. [19]. The dataset consisted of 18x2384 samples of pure olive oil and 8 times fried olive oil obtained by FTIR. The distribution of samples was equal for both classes. In the first application, the entire dataset was used for classification and regression, while in the subsequent application only the 18x8 samples containing the index features were used. The dataset was evaluated in two different ways for training and testing. In the first application, training and test data were taken half and half (Training 50% - Test 50%). In the second application, training and test data were separated by cross validation. The data obtained was evaluated using MATLAB R2018a software on a computer with Windows 10 Pro operating system with CPU (Intel Core i5-7400) 3.00GHz, 8GB RAM and 1TB hard disk. The working flow chart is given in figure 1.

The obtaining dataset was preprocessed and made suitable for classification and regression. First, classification and regression were performed using all values of the dataset. Then, classification and regression were performed using only index values. The dataset is separated in two ways for training and testing; First, the training and test values were separated 50%-50%, the CV-5 separation process was used for the other application. Linear Discriminant Analysis (LDA), Naïve Bayesian (NB) and Ensemble Tree (ET) were used as classification methods. Accuracy, F score, G_mean, Recall and Precision were used as performance measures. Gausian Process Regression (GPR) was used as the regression method and root score mean error (RMSE) was used as the performance measure. As an output estimate; 8 times fried olive oil and pure olive oil were analysed.

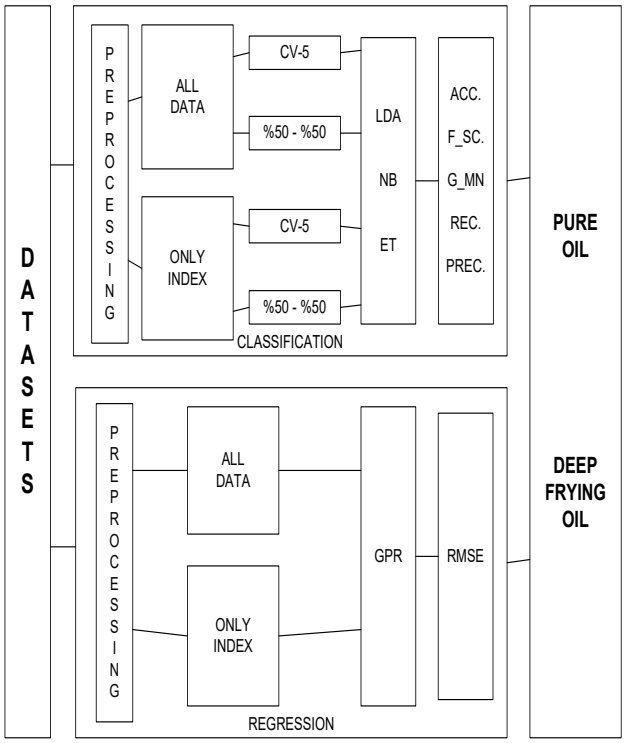


Figure 1. The working flow chart

Fourier Transform Infrared Spectroscopy

According to FTIR data obtained by Karaogul et al., The FTIR spectra acquired on a SHIMADZU-FTIR Spirit spectrometer technology with single reflection ATR accessory (Kyoto, Japan) over the wave number (400 cm -1 to 4000 cm -1) using the attenuated total reflection technique. The FTIR analysis technique was used to investigate the chemical compositions of fried oils in virgin olive oil and eight times used olive oil. The results were calculated with using Cis Index (Cs-Inx), Trans Index (Trs-Inx), Ester Index (Est-Inx), Methyl Index (Mt-Inx), Peroxide Index (Prx-Inx) and Ether Index (Et-Inx)'s equations 1-6, respectively [19-21].

$$\text{Cis lnx} = \frac{I_{722}}{I_{2854}} \quad (1)$$

$$\text{Trs lnx} = \frac{I_{913}}{I_{1460}} \quad (2)$$

$$\text{Est lnx} = \frac{I_{1743}}{I_{1460}} \quad (3)$$

$$\text{Et lnx} = \frac{I_{1159}+I_{1096}}{I_{1460}} \quad (4)$$

$$\text{Mt lnx} = \frac{I_{1363}}{I_{1460}} \quad (5)$$

$$\text{Prx lnx} = \frac{I_{3650}}{I_{1460}} \quad (6)$$

Classification and Regression

Classification and regression procedures were performed in the determination of purity of olive oil. LDA, NB and ET) were used as classification methods. LDA aims to find the projection hyperplane by minimizing the variance between classes and maximizing the distance between classes. This

hyperplane can be used for classification, dimensionality reduction, and interpretation of the importance of given features. NB is a simple and fast supervised machine learning method that uses Bayes theorem to obtain results with strong independent assumptions among the features of the data [22]. The ET enables training multiple decision tree models using the same algorithm by generating different training datasets. DT constructs a decision tree with branches and nodes based on the feature vector set. GPR method was used for regression. GPR is a non-parametric technique that occurs using a dataset to create an estimated distribution, which can average the impact of all mathematical functions that can define the phenomenon of predicting missing data points without making a predictive calculation. It requires a covariance distribution or "core function" that takes information from input data to produce accurate estimations. Core functions have hyperparameters that must be adjusted according to the properties of the input dataset [23].

Performance Evaluation

Accuracy, F score, G mean, Recall and Precission were used as success criteria for classification (eq. 7-11) [24]. Explanation of real and estimated values used in classification was shown in Table 1. For regression, RMSE success criteria were used (eq 12) [25].

Table 1. Representation of real and estimated values used in classification

		Prediction Value	
		True (1)	False (0)
Actual Value	True (1)	True Positive (TP)	False Negative (FN)
	False (0)	False Positive (FP)	True Negative (TN)

$$\mathrm{Acc} = \mathrm{TP} + \mathrm{TN} \,/\, \mathrm{TP} + \mathrm{FP} + \mathrm{TN} + \mathrm{FN}, \quad (7)$$

$$\mathrm{F-Score} = 2 \times \frac{\left(\frac{\mathrm{TP}}{FP+TN}\right)\mathrm{x}\left(\frac{\mathrm{TP}}{TP+FN}\right)}{\left(\frac{\mathrm{TP}}{FP+TN}\right)\mathrm{x}\left(\frac{\mathrm{TP}}{TP+FN}\right)}, \quad (8)$$

$$Recall = \frac{TP}{TP+FN}, \quad (9)$$

$$Precision = \frac{TP}{FP+TP}, \quad (10)$$

$$\mathrm{G-Mean} = \sqrt{\left(\frac{\mathrm{TP}}{TP+FN}\right) + \left(\frac{\mathrm{TN}}{FP+TN}\right)}, \quad (11)$$

$$\mathrm{RMSE} = \frac{\sum_{i=1}^{N}(Predicted-Actual)^2}{N}, \quad (12)$$

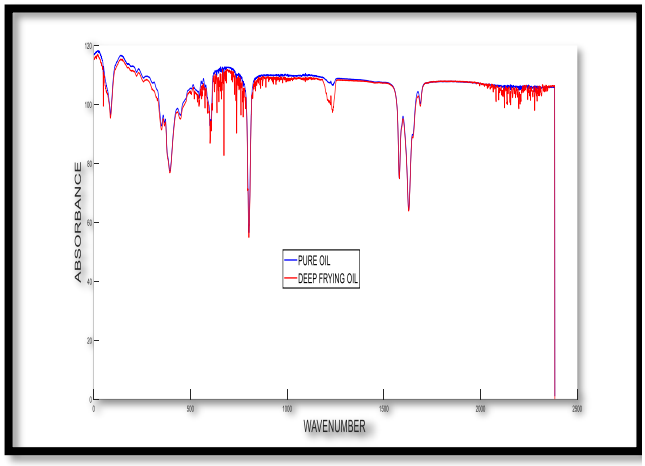
Understanding of the values given in Table 1 and Eq. 7-12; TP refers to accurately guessing the truth. (i.e. it estimated virgin olive oil as virgin olive oil). FP refers to falsely guessing the truth (Meaning that it has estimated virgin olive oil as fried olive oil). TN refers to truth guessing falsely. (i.e. it estimated fried olive oil as fried olive oil). The FN refers to truth predicting falses (On contrary to the previous outcome, meaning that it has estimated fried olive oil as virgin olive oil). Since the goal here is to predict virgin olive oil, virgin olive oils are expressed correctly and fried olive oils are incorrectly expressed. Accuracy, the ratio between correct predictions and total number of records. Recall, the number of positive patterns that are correctly classified. Precision, the number of positive patterns correctly predicted from the total predicted patterns in a positive class. [26].

Result and Discussions

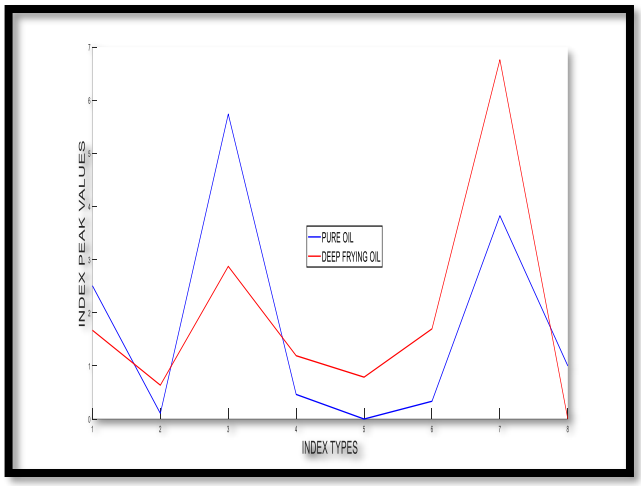
Since the raw spectra obtained as a result of spectroscopic analyzes are too complex to be evaluated visually, they can only be evaluated using chemometric data analysis methods. Chemometric models extract the information that distinguishes different groups from each other and simplify the evaluation process by excluding needless data [4]. In the study, all of the data were classified (LDA, NB and ET) in the chemometric identification method. Then, accuracy, F-score, G-mean, Recall and Precission were used as success criteria. Half of the whole data were used for training and the remaining part was used for testing. The easiest defined components were in the classification are Carbonyl peroxide, Cs-Inx, Trs-Inx, Est-Inx, Mt-Inx, Prx-Inx and Et-Inx respectively. Same dataset was used for regression. Here, the values closest to 1 could be defined more accurately and easily.

In this study, the data records of virgin olive oil and 8 times fried olive oil obtained by FTIR device were used [18]. The records obtained were evaluated by classification and regression using machine learning methods. For the evaluations and comparing for chemometric, classification and regression were made using all features in the data set. Then, the same procedures were performed using Cs-Inx, Trs-Inx, Est-Inx, Mt-Inx, Prx-Inx and Et-Inx for deep machine learning of chemical properties. In Figure 1 (a), when all properties were processed, absorbance and wavelength values of virgin and fried olive oil were given. In Figure 2 (b), index peak values and Index values of the values obtained by processing the properties that are effective in distinguishing between virgin and fried olive oil are given.

In Figure 2, the graphs shown in blue refer to virgin olive oil, while the graphs shown in red represent deep-fring oils. In Figure 2a), absorbance (y coordinate) and wavelength (x coordinate) values of virgin and deep-frying olive oils where all properties have been processed were given. In Figure 2b), Index peak values (y coordinate) and Index types (x coordinate) were given for the values obtained by processing only the properties that are effective in distinguishing between virgin and deep-fring olive oils. When the graphs were examined, it was observed that the values of virgin and deep-fring olive oils differed.



(a)



(b)

Figure 2. (a) All values of absorbance and wave number (b) For index values and index peak values

Initially, all the data were processed and the success results were compared with LDA, NB and ET from classification methods. The evaluation was performed in two stages. In the initially phase half of the dataset was used for training and the other half for testing. In the next phase, all data was used for training and testing using with CV method. The CV was divided into a data set, k discrete parts, with a total of n examples. Each time, a different set of data was allocated for testing, and the remaining k-1 data set was used for training. Each time, the test set was modified and the classifier is trained twice. The average and classifying performance of the k errors obtained in this way is estimated [26]. In this study, k=5 and n: 5768 were taken. The scores obtained are given in Table 2.

The success rates obtained using all features were compared, the values closest to 1 were expressed as the most successful scores. It was found to tbe very high value for machine learning and chemometric. This means that olive oils and DFO could be successfully interpreted by chemometric.

Table 2. All value of olive oil

Methods Used			LDA	NB	ET
Performance Evaluation	CV-5	Acc.	0,888	0,944	0,944
		F_Sc	0,875	0,941	0,947
		G_M	0,882	0,942	0,942
		Prec	1	1	0,9
		Rec	0,777	0,888	1
	%50- %50	Acc.	0.555	0,888	0,77
		F_Sc	0,5	0,888	0,75
		G_M	0,547	0,894	0,774
		Prec	0,666	1	1
		Rec	0,4	0,8	0,6

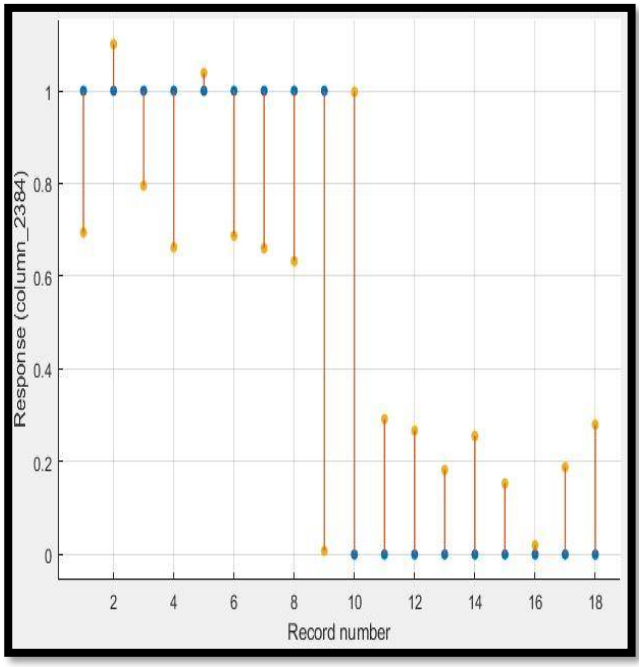
Another classification process was done using the indexes of virgin olive oil and eight times fried olive oil. The obtained scores are given in Table 3.

Table 3. For features of all index value

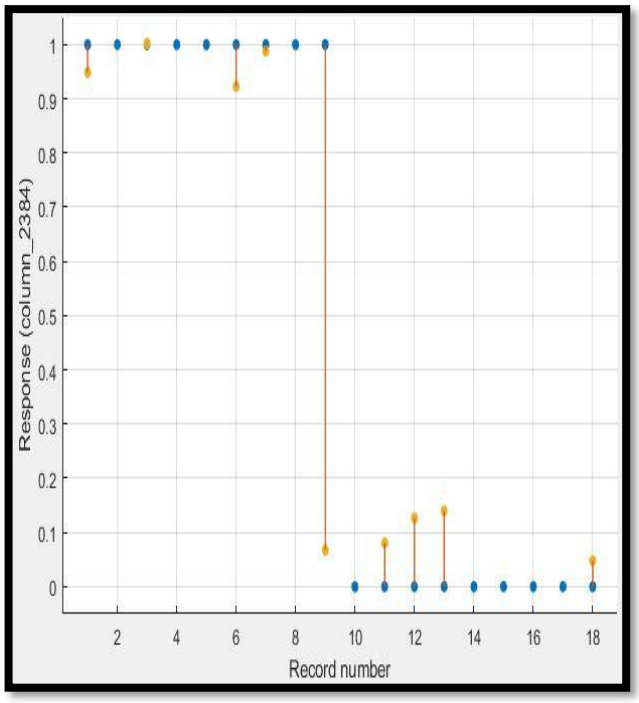
Methods Used			LDA	NB	ET
Performance Evaluation	CV-5	Acc.	0,944	1	0,944
		F_Sc	0,941	1	0,941
		G_M	0,942	1	0,942
		Prec	1	1	1
		Rec	0,888	1	0,888
	%50- %50	Acc.	0,666	0,888	0,777
		F_Sc	0,727	0,888	0,75
		G_M	0,632	0,894	0,774
		Prec	0,571	0,8	0,75
		Rec	1	1	0,75

When the results obtained in the tables are examined, the success rates obtained in the classification process using only index features were higher than in the classification process using all data. When all FTIR data were applied chemometrically, the results showed sharp differences in both virgin oil and DFO. When index data features are applied to the data set used for chemometric analysis, differences and changes in the chemical properties of oils can be observed. In addition, the efficiency rating of the index values used in the classification was made using the

relief method. The most effective features in distinguishing virgin olive oil and deep-fried olive oil was respectively carbonyl peroxide, Trs-Inx, Mt-Inx, Et-Inx, Cs-Inx and Prx-Inx.



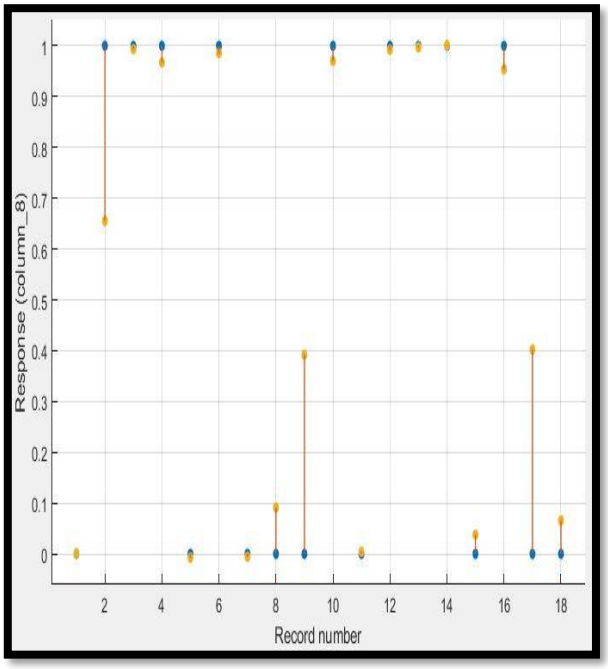
(a)



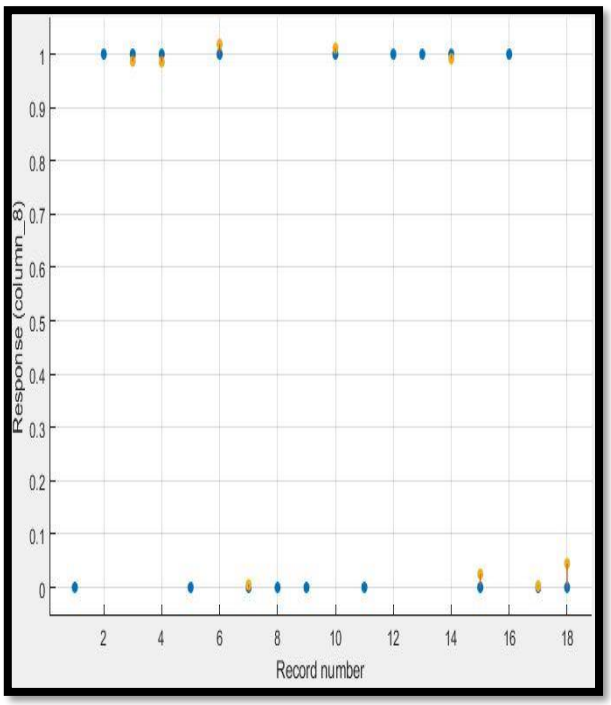
(b)

Figure 3. (a) All features CV Response (b) All features 50%-50% Response

The dataset used in the classification was also used for regression. GPR was used as a regression method and RMSE was preferred as a measure of performance. The RMSE value obtained was 0.407 when CV was used as the separation process for training and testing of the dataset. When the training and test data are equally separated, the RMSE value obtained is 0.320. For RMSE values, the values closest to 0 were expressed as the best performance. In Figure 3, response of the regression results in all features are given.



(a)



(b)

Figure 4. (a) Features of index value CV Response (b) Features of index value 50%-50% Response

Table 4. Literature Review

Techniques/ Algorithms	Performance Evaluation	Authors
Laser Induced Breakdown Spectroscopy (LIBS) and Principal Components Analysis (PCA) with Linear Discriminant Analysis (LDA) as a machine learning	Olive oil was classified 100% accurately with machine learning technique.	Bellou et al.[6]
Laser Induced Breakdown Spectroscopy (LIBS) and Absorption Spectroscopy with Linear Discriminant Analysis (LDA) and Gradient Boosting Classifier machine learning algorithms	Virgin olive oil was classified 90% accurately with machine learning technique.	Stefas et al.[7]
Laser Induced Breakdown Spectroscopy (LIBS) and Absorption Spectroscopy with Principal Component Analysis (PCA), the Linear Discriminant Analysis (LDA), the k-Nearest Neighbors (k-NN) and the Support Vector Classifiers (SVC) machine learning algorithms	Olive oil was classified 94% accurately with machine learning technique.	Gyftokostas et al.[8]
Liquid Chromatography Compled to High Resolution Mass Spectrometry (LC-HRMS) methodology and optical Spectrometry (Visible Absorption, Fluorescence and Raman) with Time aligned Region complete eXtraction (T-ReX) algorithm.	Greece virgin olive oil was classified high accuracy.	Drakopoulou et al.[10]
Gas-Chromatography-Mass-Spectroscopy, Near-Infrared Spectroscopy, a Low-Cost Electronic Nose and Machine Learning Modelling	The results showed high accuracy.	Gonzalez Viejo and Fuentes[11]
Fluorescence Spectroscopy datas with Artificial Neural Networks machine learning modelling	Olive oil's quality classified 94% accurately.	Venturini et al.[12]
Gas Chromatography (GC) with Relief Method, classified with the Support Vector Machine (SVM) and the k-Nearest Neighbor (k-NN) and Decision Tree (DT) algorithms,	The highest accuracy value for classification was calculated as 0.982 %.	Yakar and Karadağ[9]
Low-Field Nuclear Magnetic Resonance (LF-NMR) datas with machine learning approaches (Decision Tree, k-Nearest Neighbor, Linear Discriminant Analysis, Support Vector Machines And Convolutional Neural Network (CNN))	Degree of purity of Olive oil was classified with 89.29% accurately.	Hou et al.[13]
Laser Induced Breakdown Spectroscopy (LIBS) is used assisted by machine learning algorithms	Classification rates resulting in accuracies ranging between 90 and 99.2 %	Gazeli et al.[14]
Raman Spectroscopy, Principal Component Analysis (PCA), machine learning	Accuracy of 96.7% in detecting oil type	Zhao et al.[15]
Principal Component Analysis, Artificial intelligence model of the XGBoost machine learning algorithm	The highest balanced accuracy value was calculated as 99 %.	Skiada et al.[16]
Gas Chromatography, Deep Learning techniques	Accuracy value was calculated as 95 %	Vega-Marquez et al.[17]
BayesNet, Naive Bayes, Multilayer Perception, IBK, Kstar, SMO, Random Forest, J48, LWL, Logistic Regression, Simple Logistic, LogitBoost algorithms	Accuracy value was calculated as 93.88 %	Gumus et al.[18]
Fourier Transform Infrared spectroscopy (FTIR), Lineer Discriminant Analysis, Naïve Bayesian and Ensemble Tree	Olive oil was classified 100% accurately.	In this work

Using the indexed features of the same data set, regression was performed with the GPR method. The success rate of all performance criteria for GPR methods was obtained as 1 at the highest level. The RMSE obtained, when the data set was parsed with CV, was 0.158 and when half of the dataset was parsed for training and half of the dataset was tested, the obtained RMSE value was 0.031. The response and predict graphs of the regression results of the indexed features are given in Figure 4.

In the graphs shown in Figures 3 and 4, the expressions shown with the blue dots show the places where they should be (ie, the lines), and the expressions with the yellow dots show the predicted places. The lines drawn between the blue and yellow dots are drawn to visualize how far the predicted values are from the actual values. As a result of comparing the results obtained and examining the graphs, it was observed that the results using indexed features were higher when the regression results using all features and indexed features were compared.

Similar studies on olive oil, the methods used and the success results obtained are given in Table 4.

Comparison of similar studies on the subject and the applied methods with this study showed that the results obtained were usable.

Conclusions

In this study, the chemical differences of virgin oil and DFO were investigated by chemometric methods. The all wave length data of FTIR spectra and the characteristic index values of oil were theched to machine for chemometric. The chemometric methods were easier and more convenient and faster to separate of component of virgin oil and DFO with classification using machine learning methods. The multivariate method was used as a powerful tool for monitoring the samples. Chemometric models were constructed with carbonyl, peroxide, trans, methyl, ether, ester, unsaturated peroxide and cis indicex. The described calibration models were linear, suitable and precise when the contents of all index were assayed in samples. Quantification model showed high accuracy and stability. The obtained results also approve that the method is exceedingly convenient for the intended purpose. The high results obtained by the classification methods used indicate that all the indexes obtained from the FTIR right parameters and could be distinquish easily between virgin oils and DFO. This approach could be potential represents a quickly and more correct for laboratory study. Finally, enrichment of the present work with samples from more OO cultivarsis a promising near-term research prospect that will permit the generalization of the current model.

Ethics Committee Approval

There is no need to obtain permission from the ethics committee for the article prepared. There is no conflict of interest with any person / institution in the article prepared.

Authors' Contributions

The contribution of the authors is equal.

Acknowledgement

Thanks to all authors included in the article.

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