FT-NIR spectra analysis and processing to determine the quality parameters of various edible oils and chicken fat

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Valorisation of chicken fat as a fat source in chicken meat products or as a low-cost source of biodiesel could be a viable option for the poultry industry's long-term sustainability and pollution reduction. The acidity and peroxide levels of culinary oils and fats are important grading and safety factors. FT-NIR techniques with chemometric treatment are a rapid, reliable, and convenient alternative to wet-chemical characterisation by reference analysis. This research demonstrated that using FT-NIR spectroscopy (1122-902 nm) and (1090-898 nm) with PLS-R, PCA, and Discriminant Analysis (DA) was sufficient to analyse data, predict, and discriminate edible oils and chicken fat according to their quality parameters regardless of whether they are present in low or high amounts. The PLS-R regression models can predict FFA and PV because they have a perfect agreement with reference analysis (R^2, 0.94 and 0.99) and have RPD >2 showing FT-NIR is suitable for quality control applications of edible oils and chicken fat. DA was able to discriminate between the groups chicken fat and virgin olive oil, from other edible oils with a 98% accuracy, based on their FFA and PV by both methods. The FT-NIR method with a multivariate approach is an excellent alternative to reference methods, using a small sample and no chemical, fast, reliable, and as green technique that could be used as a quality control tool for both predictions of quality and discrimination purposes.

Keywords: FT-NIR, Reference Analysis, Quality parameters, FFA, PV, Multivariate methods, PCA, Discriminant Analysis

INTRODUCTION

Edible oils and fats are obtained from the extraction of oilseeds (peanut, soybean, sunflower, and so on), fruits (coconut, olive, and palm) or animal tissues. Oils and fats are used mainly for edible purposes, as ingredient or raw material, and additive in food and feed production to improve the quality and taste and to provide essential nutrient and energy, consumed as human food. Edible oils and animal fats are utilised as fat spreads, cooking fats, frying oils, salad oils, mayonnaise etc., either directly or after proper modifications. The remaining minor parts of oils and fats are processed into a variety of oleo chemicals, which are utilised as surfactants, used in pharmaceutical industries, used as animal feed and as a biodiesel [1, 2]. Chicken fat creating an environmental problem is usually considered as waste and thus it is discarded. However, it can be an alternative to edible oils and fats in food processing and can contribute to the development of a sustainability of poultry industry [3, 4]. Chicken fat can be used to increase plasticity when mixed with other solid fats [3] and can be converted to biodiesel. Oils and fats may have differences in their qualities that significantly affects their stability, reactivity, and processing. Monitoring changes and quality of oils and fats during processing and storage is very important from a quality, functionality, economic value, and food safety point of view. The nutritional value,

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Received: August 22, 2022
Accepted: October 11, 2022

freshness and quality of edible oils greatly affected by acidity and peroxide value that also affects human health and may cause problems during processing [5]. Acidity is determined by acid/base titration and may change during storage, processing, heating, or frying due to time, temperature, and moisture content. Besides, the acidity of edible oils shows hydrolysis or lipolysis, thus it is a direct measure of the quality [6] and tendency for rancidity. Oils and fats containing high amounts of free fatty acids (FFAs) are more prone to oxidation and produce rancidity, since FFAs are less stable, and thus it greatly affects quality and commercial value of oils and fats [7] as well as for their classification [8]. FFA content of edible oils and fats are reduced during refining and biodiesel production [4, 7]. The peroxide value (PV) being normally determined by titration is an indicator of freshness, it reflects oils oxidative level and thus its tendency to become rancid and therefore, it is a very important quality parameter for food safety [9]. Oxidative degradation generates a negative impact on flavour, shelf life, and nutrition of oils and fats [10, 11]. Peroxide value is below 10 meq O2/kg for fresh oils and if PV as high as 100 meq O2/kg might be the cases of food poisoning [10]. The American Oil Chemists Society (AOCS) and International Olive Council (IOC) have recommended standard titrimetric methods for measuring FFA and PV of oils and fats [12, 13]. These titrimetric methods have some disadvantages since they are time-consuming, laborious, tedious and can result in health and environmental problems, inconvenient for on-line monitoring, expensive, poorly reproducibile, and less sensitive [5, 7, 14-16]. These chemical methods require large amounts of organic solvents, toxic and carcinogenic reagents that cause health concerns and environmental disposal problems and difficulty in distinguishing the end-point with dark coloured oils and fats and largely dependent on the skills of the analyst [5, 11]. Therefore, reliable, fast and safe analytical methods are required to determine quality parameters of oils and fats due to differences in composition, production, refining, blending, or adulteration [16] that should be addressed by the official authorities and producers [16, 17]. The Fourier transform Near infrared (FTNIR) technique combined with chemometrics has been developed as an analytical tool for determination of oils and fats quality. It is a rapid technique (takes few minutes) and reduces the use of toxic solvents, pollution-free, safe to use, helps environmental protection, it is economical, a simple operation even for untrained staff, efficient and allows online, off-line and at-line detection of quality parameters for use of quantification of various oil parameters including acidity based on C-H stretching and peroxide value based on COO stretching [2, 5, 11, 18] . The FT-NIR technique could determine and predict several parameters such as acidity, peroxide value, iodine value, anisidine value, malondialdehyde, soap contents within a single measurement [6,7,18-20] providing a great amount of information which is useful for determination of quality of oils and fats. However, NIR spectra have wide and overlapping bands due to the similar nature of oils and fats, therefore needs chemometric methods such as principal component analysis (PCA) and Partial Least Square regression (PLS-R) need to be used to detect spectral differences by computing latent variables, known as loadings spectra, that are related to the component of interest for evaluation of data and interpretation of quality parameters [21, 22, 23]. Thus, chemometric analysis methods has been frequently developed for the rapid and online FT-NIR spectroscopic detection system for food quality, safety and control and has been used for the discrimination of edible oils and fats [19, 20], classification [16] and to distinguish animal fats from different species [1]. There are many uses for FTNIR spectroscopy in determining the origin of edible oils and performing general analysis in edible oils and fats. Putri et al. [5] tested some quality parameters like acidity, peroxide, and saponification values in patin fish oil with the FTIR spectroscopy combined with Principal Component Regression (PCR) and (PLS-R) providing a high correlation coefficient (R^2) reached up to 0.99 with FT-NIR range 721 to 2950 cm-1. Galbraith et al. [10] used NIR to build regression models to predict and determine peroxide value of the various edible oils within NIR range of 3799-14,998 cm^{-1} and RMSEP ranged between 1.9 to 2.50. Jiang et al. [17] was able to have excellent performance in predicting acid value of edible oils during storage with MPA based strategy, R^2 = 0.92 and RPD was 2.82 by NIR with a range of 1150-1700 nm. Also, Kaufmann, et al. [23] have used PLS calibration model for acidity prediction in palm oil, achieving R^2 = 0.97 using most relevant wavelengths range of 1,100 to 1,500 nm. Thus, previous studies consisted of quality parameters such as acidity [6, 7, 17, 21, 23] and peroxide values [5, 9, 11, 20] of single type oils and fats. To best of our knowledge, the FT-NIR method has not been applied for determination of the free acidity and peroxide value to assess chicken fat quality. Therefore, the aim of this study is to investigate, compare and highlight the potential of the FT-NIR spectroscopic techniques to monitor free acidity and peroxide values of various common edible oils and chicken fat both by reference analysis and FT-NIR technique and to construct a reliable multivariate model to predict and discriminate edible oils and chicken fat according to their quality parameters.

2. MATERIALS AND METHODS

2.1. MATERIALS

Sunflower oil, corn oil, virgin, and the olive oil with three different brands were purchased from local markets in 1 kg/bottle and chicken fat was provided from Pilyem Feed factory, Turkey. All edible oils and

chicken fats were stored at 5±1°C, respectively in the dark until the related analysis.
The chemicals and solvents used throughout the study were HPLC grade. N-hexane, chloroform, ethyl acetate, ethanol, HCl, acetic acid, sodium thiosulphate, KI, acetone, KOH, and phenolphthalein were obtained from Merck (Darmstadt, Steinheim Germany).

2.2. METHODS

2.2.1. Analytical Measurements

The free acidity as a percentage of oleic acid (% w/w) and the peroxide value as meq O_2 kg^{-1} were determined, according to Ca 5a-40 and Cd 8-53, respectively, described in American Oil Chemist Society [12] official reference methods.

2.2.2. FT-NIR Spectroscopy

The FT NIR spectra of edible oils and chicken fat were measured as indicative of quality parameters of edible oils and chicken fats. The spectrophotometer was a Multi-Purpose Analyzer (MPA) Fourier Transform Near Infrared Transmittance FT-NIR (Bruker Optics, Ettlingen, Germany) fitted with an inGaAs detector and thermostated between 5 and 35°C. The FT-NIR spectra were acquired with a 10 kHz scanner velocity from 12500 (2500 nm) to 4000 (800 nm) cm^{-1}, with 5 scans per spectrum and an 8 cm^{-1} resolution. In 30 seconds, the entire sample FT-NIR spectrum was captured. Chicken fat was heated to 50°C to guarantee that it was completely melted, translucent as described in [21] before scanning. The cell components were washed in warm water, rinsed with acetone, and dried after each sample. Each edible oil and chicken fat spectra was collected in triplicate. OPUS program fully GMP compliant, fully 21 CFR part 11 compliant from Bruker was employed for data acquisition. Treatment of data OPUS/Quant 2 was used to carry out the NIR calibration process (Bruker Optics GmbH, Ettlingen, Germany). Multiple components can be quantified within a single spectrum using software applications.

2.2.3. Chemometric Analysis

The mean and standard deviation of three measurements were used to calculate the results. Oils and fat FT-NIR spectra in the range of 12.500 (800 nm) –4.000 (2500 nm) cm^{-1}. A paired sample t-test ($p<0,05$) and z-score was used to compare the data obtained by reference and FT-NIR method. PCA-Correlation, PLS-Regression, and Discriminant Analysis (DA) were used to assess the quality by both official reference and FT-NIR methods using XLSTAT 2022.1.1.1251, Addinsoft, New York, NY, USA software package.

2.2.4. Data Analysis (PCA), Model Performance (PLS-R) and Discrimination (DA)

PCA was used initially to examine the possible classification of the various edible oils and fats with a full correlation since it enables reducing variable dimensions for samples clustering. The first principal component, PC1, covers the maximal information direction and is orthogonal (that is, explains complementary information) to PC2. PCA is an unsupervised exploratory method that is linear combinations of the original variables. If they are closer they are more similar, if they are further apart they more distinct in the score plots. Thus, the plots can be used to deduce sample differences and similarities. Samples to the right of the scores plot, for example, will often have a large value for variables to the right of the loadings plot and a small value for variables to the left of the loadings plot [18]. The model performances FT-NIR-PLS-R models of edible oils and chicken fat were evaluated based on determination of correlation coefficient R^2, RMSE and RPD values of the calibration models of PLS-R. The R^2 is an indicator of the goodness of fit between the predicted and reference values for each quality parameter (free acidity and peroxide value) and it may change between 0 and 1 indicating fitness of the models [14, 21]. The ratio of the standard deviation of the reference data divided by the standard error of prediction is known as RPD (Ratio of Performance Deviation) is also used to check the accuracy of the prediction models that have been constructed. According to extensive research, a PLS model with an RPD value between 2.0 and 3.0 is regarded as a decent PLS model and adequate for analytical purposes [14]. The Discriminant Analysis (DA) model was built by using the backward stepwise analysis option (within-class covariance matrices are assumed to be equal) was performed to discriminate edible oils and chicken fats according to their quality criteria.

3. RESULTS AND DISCUSSION

3.1. FT-NIR SPECTRA AND REFERENCE ANALYSIS VALUES OF FREE ACIDITY AND PEROXIDE VALUE OF EDIBLE OILS AND CHICKEN FAT

Free fatty acidity is used to constitute the quality and the classification of edible oils. Hydroperoxides are formed due to oxidation of fatty acids and are measured through peroxide value. Both are the most significant parameters to determine quality of edible oils and fats [21, 22]. FT-NIR spectra pre-processing (normalisation) it is necessary to evaluate the results by using multivariate methods for interpretation [23] and to detect spectral differences by computing latent variables (loading spectra) that are related to the free fatty acidity and peroxide value of oils and chicken fat [21]. Figure 1 shows FT-NIR spectra of chicken fat and edible oils.
FT-NIR spectra obtained at 5450 and 4490 cm^{-1} (1090-898 nm) and 5612 and 4509 cm^{-1} (1122-902 nm) and the peaks at can be appointed to the first overtones of C-H stretching and C=O stretching vi-

brations, respectively. FT-NIR spectra of edible oils and chicken fat showed that the most intense absorption bands at 5650 and 4490 cm^{-1} corresponds to free fatty acidity and peroxide value (Fig. 1). Multiple components can be quantified within a single spectrum using software applications [21, 23-25]. Edible oils and chicken fat have similar spectra due to their nature (Fig. 1). The spectra obtained between 5612 to 4500 was used for FFA and 5450 to 4489 was used for PV determinations in FT-NIR after 1st normalization process in this study. The reference titrimetric method data was incorporated into the Bruker FT-NIR system to establish a spectral library for the creation of a quick, non-destructive approach to test oils and chicken fat quality. Table I shows the FFA and peroxide values of edible oils and chicken fat that are determined both by reference and NIR methods.

The results showed that there were variations in the free fatty acid and peroxide value contents of oils and fats obtained from three different brands (Tab. I). The free fatty acid level of the edible oils and fats ranged between 0.28 (corn oil) to 9.63 (chicken fat) % (as oleic acid) determined both by reference analysis and FT-NIR method, respectively. It was found that the virgin olive oil had the highest and corn oil had the lowest peroxide value of 17.91 and 11.48 $meqO_2$ $kg^{-1}$of determined by both reference analysis and FT-NIR method, respectively (Tab. I). According to Table I p-value is 0.05 and edible oils did not exhibit significant differences in PV values between reference and FT-NIR method showed in FFA content of corn oil and chicken fat ($p<0,05$). Z-scores were between -0.13 and -0.17 respectively confirming the accuracy of the FT-NIR-method against reference method for FFA and PV determination. Our results agree with [22, 26, 27] found no significant differences between reference method and FT-NIR method for (fat, protein, and water) composition as well as quality parameters of oils and fats including FFA, PV.

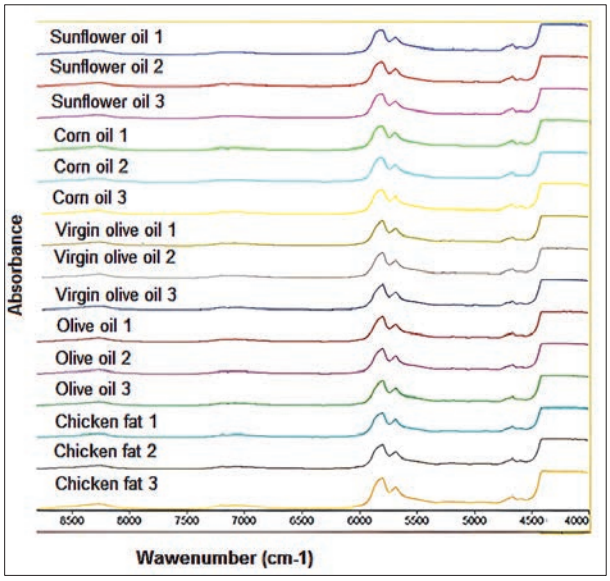

Figure 1 - FT-NIR spectrum of edible oils and chicken fat. Spectra were obtained in the transmittance mode using 6.5 mm i.d. glass vials and accumulating 5 scans per spectrum and a resolution of 8 cm^{-1}. Spectra were shifted on the y-axis to clearly show their characteristic bands. Inset: Differences between the different types of edible oils and chicken fat were analyzed in the interval from 4600 to 8500 cm^{-1} (833 - 2500 nm).

3.2. PREDICTION MODELS AND COMPARISON OF FT-NIR AND REFERENCE METHODS

FT-NIR spectroscopy coupled with is a factorial multivariate calibration method such as Partial least square

Table I - Comparison of Free Fatty Acidity and Peroxide Values of Edible Oils and Chicken Fat by Reference Titrimetric Method and FT-NIR Spectroscopic Method

Edible oils & Fats		Free Fatty Acidity (% as oleic acid)	Peroxide Value (meq O_2 kg^{-1} oil)
Chicken Fat	Reference	9.15±2.81	12.87±5.01
	FT-NIR	9.63±3.32	12.98±4.88
	p-value	0.11	0.80
Corn Oil	Reference	0.28±0.14	12.87±0.76
	FT-NIR	0.46±0.28	11.48±2.13
	p-value	0.37	0.27
Olive Oil	Reference	0.59±0.29	12.84±7.18
	FT-NIR	0.74±0.36	13.67±6.35
	p-value	0.03*	0.54
Sunflower Oil	Reference	0.50±0.16	17.08±2.19
	FT-NIR	0.56±0.43	15.94±4.35
	p-value	0.00**	0.56
Virgin Olive Oil	Reference	1.31±0.17	16.80±11.16
	FT-NIR	0.99±0.19	17.91±12.45
	p-value	0.00**	0.50
	z-score	-0.13	-0.17

*<0.05 **<0.01

(PLS-R) were found to be effective in building the calibration models from variables that has been extensively used for the quality parameters of edible oils [28-30]. The PLS-R models using the FT-NIR spectra for the prediction of the content of the free fatty acids and peroxides found in edible oils and chicken fat was developed by using cross validation (Jacknife), standardised coefficient models. Correlation plots and PLS-R model data are given in Figure 2a-b.

FT-NIR spectra were evaluated by using PLS-R calibration models to correlate and predict free fatty acidity and peroxide values for both official reference analysis and FT-NIR method. Although there is a clear difference between FFA content of chicken fat (9.6%, the highest) and corn oil (0.3%, the lowest) determined by both method, good regression models could be obtained through PLS-R regression, cross validated models by using standardised coefficients. This clearly indicates that both models used for determining and predicting the values of free fatty acidity and peroxide values of edible oils and chicken fat are acceptable models to be predicted or measured accurately by using the FT-NIR Method no matter whether they are present in high or low amounts.

PLS-R regression for free fatty acidity and PV in edible oils and chicken fat evaluated by PLS-R-FT-NIR and official reference method provided equations were as follows; FFA (FT-NIR) = -0,027+0,55* REF FFA with correlation coefficient R^2 = 0.99, RMSE = 0.35 and RPD = 3.1 and PV (FT-NIR) = -0,89 + 0,52* REF PV with correlation coefficient R^2 = 0.94, RMSE = 1.57 and RPD = 2.4, respectively. Our results clearly showed that good prediction models can be developed using the PLS-R technique for the prediction of quality parameters of edible oils and chicken fat since R^2 values were very close to 1 (0.99 and 0.94, respectively) (Fig. 2a, b). PLS regression models can result in accurate models even though the constituents concentrations vary [31] for different types of oils used [10]. While FFA values range between 0.3 to 9.6% and peroxide values between 11.5 to 17.9 meq O2/kg, RMSE values vary between 0.35-1.57. These results clearly show a good prediction models for FFA and PV were obtained from FT-NIR and reference methods. Similar results were also obtained by [31] FFA values ranged between 0.27-11.70%, RMSE values were obtained as 0.47 and 0.61. Our results were in accordance with the previous studies estimated with similar R^2 values found between 0.85 to 0.99 and 0.81 to 0.99 [14, 22, 24, 32, 33] for FFA and peroxide values of olive oil and other edible oils. The RPD values for free fatty acidity and PV values of edible oils and chicken fat were found between 3.1 and 2.4 respectively. This clearly indicates, in addition to R^2 values, that both models used for determining and predicting the values of free fatty acidity and peroxide values of edible oils and chicken fat are acceptable models and free fatty acid and peroxide value of the oils and chicken fat can be predicted accurately by using the FT-NIR method compared to the reference method. In fact, it may be difficult to obtain RPD values higher than 3, because of the sample preparation, presentation, or difficulty with reference testing, and a sample set with minimal variability. RPD > 3.0

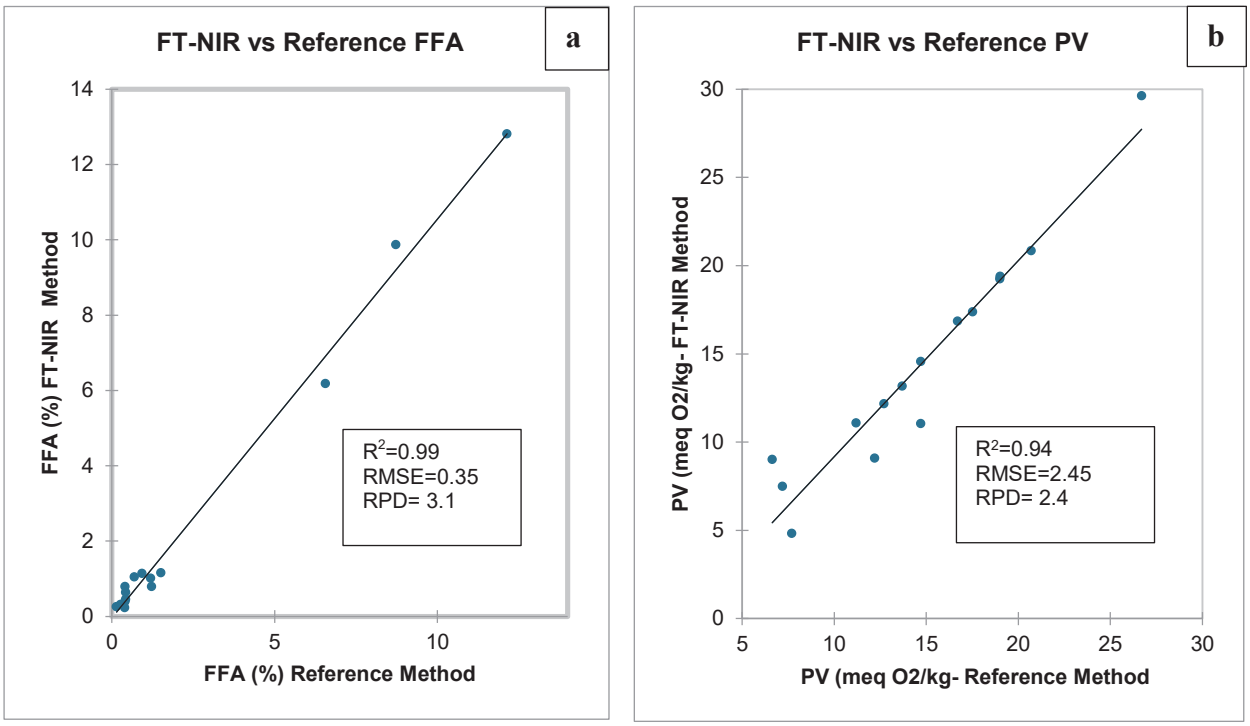

Figure 2 a-b - (a) Correlation plots and PLS-R modeling for Reference and-FT-NIR Method for (a): free fatty acidity-FFA (% as oleic acid), (b): peroxide value-PV (meqO2 kg^{-1} of oil) of edible oils and chicken fat.

can be used for screening quality, quality control, process control and high enough for reliability and prediction, RPD > 8 suitable for any application [18, 21, 25, 31, 32, 34] and RPD higher than 10 is considered equivalent to the reference method [35]. Thus, our results agree with previous studies, where the FT-NIR spectroscopy was used to predict edible oils quality parameters by using PLS-R models based on their R^2 and RPD values [14, 17, 33, 30]. This study showed that FFA and PV of edible oils and chicken fat were determined accurately by using the FT-NIR method. These quality indicators can be accurately predicted by utilising PLS-R methods. Because accuracy and repeatability were excellent, and the measurement time was only approximately 30 seconds per sample, the FT-NIR approach could be useful for determining FFA and PV of edible oils and chicken fat.

Similar results were also confirmed by correlation tests (data not shown). The correlation results clearly indicated that official reference methods and the corresponding FT-NIR methods confirms that FT-NIR is very strong tool to analyse edible oils and fat quality parameters. This method is a simple and convenient way to check quality, with the benefits of ease of use, quick sample turnover, and no sample pre-treatment. In terms of analytical performance, the results of multivariate aided FT-NIR analysis were statistically like those produced by official and traditional processes. Thus, this technique, could be applied for the quality control, safety evaluation and discrimination of different edible oils and fats, it reduces time, costs, and the possible chemical hazard of reference analysis. Successful prediction of ripening degree and phenolic compounds, chlorophyll content, essential oil of olives and olive oil, oregano oil and calila oil leaves [33, 36-38], FFA, PV, total phenolic content and fatty acids of oils [10, 18, 33] are a few examples of FT-NIR approaches being used in prediction research in literature.

3.3. DISCRIMINATION OF EDIBLE OILS AND CHICKEN FAT BY BOTH FT-NIR AND REFERENCE METHODS

The PCA score biplot of FFA and PV results of edible oils and chicken fat both by reference and FT-NIR method is shown in Figure 3a.

Various oils and chicken fat which were analysed with FT-NIR spectra were classified and clustering tendencies were determined by using the PCA method in literature [14, 24, 39].

PC1 was mainly correlated with the free fatty acids and PC2 was correlated with peroxide values determined by both methods. The first principal component (PC1) explains 64.3% of the total variance, and the second major component (PC2) represents 32.3% of the total variance. Edible oils show a negative contribution to PC1 and chicken fat that have the high free fatty acidity (9.6%) were positioned on the positive PC1 axis. The PCA graphs clearly demonstrated that chicken fat shows a significant difference than other edible oils (Fig. 3a). It was clear that PCA clustered oils and chicken fat into three groups of chicken fat highly and positively, sunflower oil and vir-

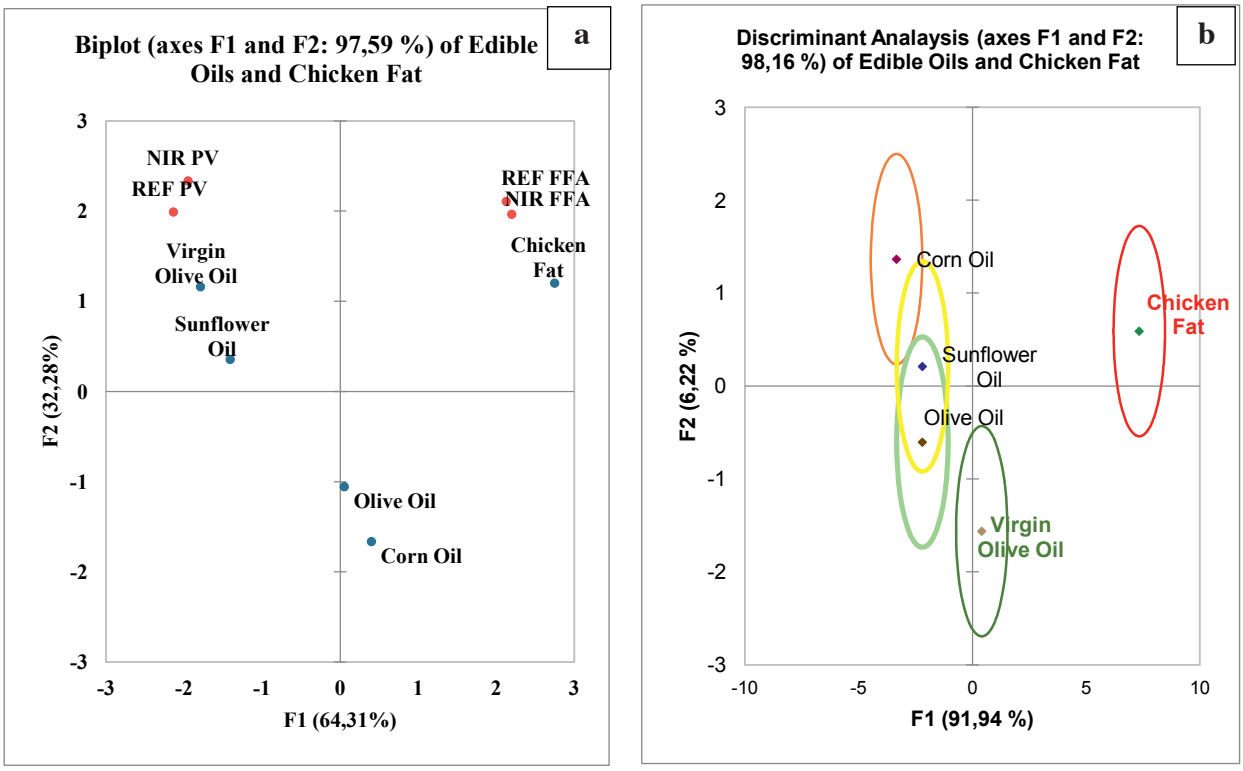

Figure 3 a-b - (a) Bioplot of first two principal components of PCA, (b) Discriminant Analysis (DA) of edible oils and chicken fat

gin olive oil are highly and negatively correlated with PC1 and corn oil, the olive oil are highly and negatively correlated with PC2 (Fig. 3a).
The discrimination of edible oils and chicken fat was achieved by DA analysis (Fig. 3b). The first two discriminant functions explained 98.16% of the total variance, according to the groups in the score plot for oils and chicken fat. Chicken fat and virgin olive oil were separated clearly from olive oil, sunflower oil and corn oil, and there are overlapping between sunflower and olive oil (Fig. 3b). The results clearly showed that good quality groupings were achieved by DA for edible oils and chicken fat. This study showed that using FFA and PV as quality criteria, FT-NIR with chemometric treatment could correctly distinguish virgin olive oil from olive oil, sunflower oil, corn oil by discriminant analysis.
This separation/discrimination of chicken fat could be due to its fatty acid composition and oxidative stability) and the highest concentration of FFA as compared to edible oils (Tab. I). Thus chicken fat and virgin olive oil could be discriminated from edible oils and fats based on FFA and PV values by using both reference method and FT-NIR methods with DA. Our results were consistent with the results of [16, 40] that could discriminate lard, butter from vegetable oils based on iodine values by using PCA, PLS-DA, DA and canonical variate analysis.
Our results are consistent with the results of [14, 24, 40] that managed to discriminate oils and [1, 26] chicken fat and animal fats based on quality parameters and fatty acid composition. [41] clustered the oils according to acidity and peroxide index manage to cluster olive oils from sunflower seed and corn oil and [14] separated olive oils depending on their grade as extra virgin, virgin, ordinary virgin and lampante oils [24] due to their own unique clustering trends linked with their storage durations, oils like soybean oil, rapeseed oil, corn oil, and sunflower seed oil were separated [40] clustered pure and adulterated palm oil samples according to PCA graph. Recently, [26] easily discriminated lard as animal fat from vegetable oils by using FAMEs as metabolomics with chemometric treatment.

4. CONCLUSIONS

The monitoring of acidity and peroxide value is very important for quality and the safety of edible oils and chicken fat. The official reference method for determining free fatty acidity and peroxide values in edible oils and chicken fat is tedious, time-consuming, arduous, tiresome, and damaging, and it is not suitable for online use. The results of study clearly showed that FT-NIR spectroscopy (4500 to 5600) was satisfactory to determine and predict free fatty acidity and peroxide contents of edible oils and chicken fat with a good correlation (94 to 99%) with reference analysis (R^2 = 0.99 and 0.94, RPD > 2) and to discriminate edible oils and chicken fat based on their acidity and peroxide value. Chicken fat and virgin olive oil was discriminated from other edible oils with a 98% accuracy, based on their FFA and PV. FT-NIR method with chemometric treatment could be used for quality control and prediction and discrimination purposes as a convenient, green, fast, and accurate alternative to reference titrimetric methods. It gives government authorities and stakeholders a useful tool for assessing the quality of culinary oils and chicken fat quickly. This method can be a time and solvent-saving option for routine analysis of a large number of oils and fats samples, particularly for high throughput results during industrial processing that allow in-process optimisation of technological parameters. Further research in this field with other quality and purity parameters for oils and fats is needed to confirm the possible application of FT-NIR with multivariate approaches for quality assurance and safety of the oils and fats.

Funding and acknowledgements

This research was funded by Scientific Research Projects Unit of Cukurova University, Turkey with Project No: FYL-2019-12342. The authors wish to acknowledge Cukurova University for financial support. This manuscript is prepared from the MSc thesis of Askim Akca Mecit.

REFERENCES

[1] F. Gao, D. Ben-Amotz, S. Zhou, Z. Yang, L. Han, X. Liu, Comparison and chemical structure-related basis of species discrimination of animal fats by Raman spectroscopy using near-infrared and visible excitation lasers, LWT, 13. 110105 (2020).
[2] X. Li, J. Zhao, L.H. Liu, J. Tan, Optical properties of edible oils within spectral range from 300 to 2500 nm determined by double optical pathlength transmission method, Applied Optics 54, 3886-3893, (2015).
[3] L.M. Peña-Saldarriaga, J. Fernández-López, J.A. Pérez-Alvarez, Quality of Chicken Fat by-Products: Lipid Profile and Colour Properties, Foods 9, 1046 (2020).
[4] V.A.M. Selvan, N.K. Gurusala, A. Balan, S. Athreya, Optimization of Waste Chicken Fat Pre-Treatment Process for Biodiesel Production, Transactions of SAE, 2013 (2014).
[5] A.R. Putri, A. Rohman, W. Setyaningsih, S. Riyanto, Determination of acid, peroxide, and saponification value in patin fish oil by FTIR spectroscopy combined with chemometrics, Food Research 4, 1758-1766 (2020).
[6] I. Tarhan, A.A. Ismail, H. Kara, Quantitative determination of free fatty acids in extra virgin olive oils by multivariate methods and Fourier transform infrared spectroscopy considering different

absorption modes, International Journal of Food Properties 20, S790-S797 (2017).
[7] S.A. Mahesar, S.N. Shah, A.W. Mahesar, A.A. Kandhro, A.R. Khaskheli, P. Menghwar, S.T.H. Sherazi, A chemometric approach for the quantification of free fatty acids in cottonseed oil by Fourier transform infrared spectroscopy, International Journal of Food Properties 20, 1913-1920 (2017).
[8] A. Kiritsakis, T. M. Keceli, K. Kiritsakis, Olive Oil, in: F. Shahidi (Ed.) Bailey's Industrial Oil and Fat Products, 1-38, Wiley, Co. (2020).
[9] A.M. Marina, Quantitative Analysis of Peroxide Value in Virgin Coconut Oil by ATRFTIR Spectroscopy, The Open Conference Proceedings Journal 4, 53-56, (2013).
[10] W.E. Gilbraith, J.C. Carter, K.L. Adams, K.S. Booksh, J.M. Ottaway, Improving Prediction of Peroxide Value of Edible Oils Using Regularized Regression Models, Molecules 26, 7281 (2021).
[11] X.Yu, Q. Li, D. Sun, X. Dong, T. Wang, Determination of peroxide value of edible oils by FTIR spectroscopy using polyethylene film, Anal. Methods, 7, (2014).
[12] AOCS, AOCS Official methods and recommended practices of the American Oil Chemists' Society III. AOCS (Method Cd 8-53 and Ca 5a-40), in, AOCS, Campaign, IL, (2017).
[13] IOC, International Olive Council. Trade Standard Applying to Olive Oils and Olive Pomace Oils. COI/T.15/NC No 3/Rev. 16 June 2021, in: COI/T.15/NC No 3/Rev. 16 June 2021, International Olive Oil Council, Madrid, 1-18, (2021).
[14] N. Abu-Khalaf, M. Hmidat, Visible/Near Infrared (VIS/NIR) spectroscopy as an optical sensor for evaluating olive oil quality, Comput Electron Agr, 173 (2020).
[15] L. Dymińska, M. Calik, A.M.M. Albegar, A. Zając, K. Kostyń, J. Lorenc, J. Hanuza, Quantitative determination of the iodine values of unsaturated plant oils using infrared and Raman spectroscopy methods, International Journal of Food Properties 20, 2003-2015 (2017).
[16] P. Samyn, D. Nieuwkerke, G. Schoukens, L. Vonck, D. Stanssens, H. Aabbeele, Quality and Statistical Classification of Brazilian Vegetable Oils Using Mid-Infrared and Raman Spectroscopy, Appl Spectrosc 66, 552-565 (2012).
[17] H. Jiang, Y.C. He, Q.S. Chen, Determination of acid value during edible oil storage using a portable NIR spectroscopy system combined with variable selection algorithms based on an MPA-based strategy, J Sci Food Agr 101, 3328-3335 (2021).
[18] E.A. Seweh, Z. Xiaobo, F. Tao, S. Jiachen, H.E. Tahir, M. Arslan, Multivariate analysis of three chemometric algorithms on rapid prediction of some important quality parameters of crude shea butter using Fourier transform-near infrared spectroscopy, J near Infrared Spec 27, 220-231(2019).
[19] L.S. Vieira, C. Assis, M.E.L.R. de Queiroz, A.A. Neves, A.F. de Oliveira, Building robust models for identification of adulteration in olive oil using FT-NIR, PLS-DA and variable selection, Food Chem, 345 (2021).
[20] A. Rohman, M.A.I.B. Ghazali, A. Windarsih, I. Irnawati, S. Riyanto, F.M. Yusof, S. Mustafa, Comprehensive Review on Application of FTIR Spectroscopy Coupled with Chemometrics for Authentication Analysis of Fats and Oils in the Food Products, Molecules 25, 5485 (2020).
[21] P. Adewale, O. Mba, M.-J. Dumont, M. Ngadi, R. Cocciardi, Determination of the iodine value and the free fatty acid content of waste animal fat blends using FT-NIR, Vib Spectrosc 72, 72-78 (2014).
[22] I. Willenberg, B. Matthaus, C. Gertz, A New Statistical Approach to Describe the Quality of Extra Virgin Olive Oils Using Near Infrared Spectroscopy (NIR) and Traditional Analytical Parameters, Eur J Lipid Sci Tech, 121 (2019).
[23] K.C. Kaufmann, F.D. Favero, M.A.M. de Vasconcelos, H.T. Godoy, K.A. Sampaio, D.F. Barbin, Portable NIR Spectrometer for Prediction of Palm Oil Acidity, J Food Sci 84, 406-411(2019).
[24] Y.C. He, H. Jiang, Q.S. Chen, High-precision identification of the actual storage periods of edible oil by FT-NIR spectroscopy combined with chemometric methods, Anal Methods-Uk, 12 3722-3728 (2020).
[25] R.K.K. Raigar, S. Srivast, H.N. Mishra, Implementation of Non Destructive FTNIR Method for Quick Estimation of Peanut Quality Based on FFA and Peroxide Value, in, Research Square Platform LLC, (2021).
[26] M. Heidari, Z. Talebpour, Z. Abdollahpour, N. Adib, Z. Ghanavi, H.Y. Aboul-Enein, Discrimination between vegetable oil and animal fat by a metabolomics approach using gas chromatography–mass spectrometry combined with chemometrics, Journal of Food Science and Technology, 573415-3425 (2020).
[27] J. Mlček, K. Šustová, J. Simeonovová, Application of FT NIR spectroscopy in the determination of basic chemical composition of pork and beef, Czech Journal of Animal Science, 51361-368 (2011).
[28] R.M. Maggio, L. Cerretani, E. Chiavaro, T.S. Kaufman, A. Bendini, A novel chemometric strategy for the estimation of extra virgin olive oil adulteration with edible oils, Food Control 21, 890-895, (2010).
[29] P. Liang, H. Wang, C. Chen, F. Ge, D. Liu, S. Li, B. Han, X. Xiong, S. Zhao, The Use of Fourier Transform Infrared Spectroscopy for Quantification of Adulteration in Virgin Walnut Oil, Journal of Spectroscopy, 1-6, (2013).

[30] I.S. Ozdemir, O. Karaoglu, C. Dag, S. Bekiroglu, Assessment of sesame oil fatty acid and sterol composition with FT-NIR spectroscopy and chemometrics, Turk J Agric For 42, 444-452, (2018).
[31] I. Dogruer, H.H. Uyar, O. Uncu, B. Ozen, Prediction of chemical parameters and authentication of various cold pressed oils with fluorescence and mid-infrared spectroscopic methods, Food Chem 345, 128815 (2021).
[32] H. Jiang, Q.S. Chen, Determination of Adulteration Content in Extra Virgin Olive Oil Using FT-NIR Spectroscopy Combined with the BOSS-PLS Algorithm, Molecules 24, (2019).
[33] J.A.C. Sanchez, W. Moreda, J.M. Garcia, Rapid Determination of Olive Oil Oxidative Stability and Its Major Quality Parameters Using Vis/NIR Transmittance Spectroscopy, J Agr Food Chem, 618056-8062 (2013).
[34] N. Prieto, O. Pawluczyk, M.E.R. Dugan, J.L. Aalhus, A Review of the Principles and Applications of Near-Infrared Spectroscopy to Characterize Meat, Fat, and Meat Products, Appl Spectrosc 71, 1403-1426 (2017).
[35] R. Riovanto, M. De Marchi, M. Cassandro, M. Penasa, Use of near infrared transmittance spectroscopy to predict fatty acid composition of chicken meat, Food Chem 134, 2459-2464, (2012).
[36] C. Alamprese, S. Grassi, A. Tugnolo, E. Casiraghi, Prediction of olive ripening degree combining image analysis and FT-NIR spectroscopy for virgin olive oil optimisation, Food Control, 123, (2021).
[37] N. Wu, J.A. Liu, G.Y. Zhou, R.K. Yan, L. Zhang, Prediction of Chlorophyll Content of Leaves of Oil Camelliae after Being Infected with Anthracnose Based on Vis/NIR Spectroscopy, Spectrosc Spect Anal 32, 1221-1224 (2012).
[38] S.P. Zhu, G. Wang, F. Yang, J.Q. Kan, J. Guo, Q.M. Qiu, Effect of powder's particle size on the quantitative prediction of volatile oil content in zanthoxylum bungeagum by NIR technique, Spectrosc Spect Anal 28, 775-779, (2008).
[39] A.S. Luna, A.P. da Silva, J.S.A. Pinho, J. Ferre, R. Boque, Rapid characterization of transgenic and non-transgenic soybean oils by chemometric methods using NIR spectroscopy, Spectrochim Acta A, 100, 115-119, (2013).
[40] E. Teye, C. Elliott, L.K. Sam-Amoah, C. Mingle, Rapid and nondestructive fraud detection of palm oil adulteration with Sudan dyes using portable NIR spectroscopic techniques, Food Addit Contam A 36, 1589-1596, (2019).
[41] S. Armenta, S. Garrigues, M. de la Guardia, Determination of edible oil parameters by near infrared spectrometry, Anal Chim Acta 596, 330-337, (2007).