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Corn Oil Authentication Using ATR-FTIR Spectroscopy and Chemometrics

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Abstract

The authentication of edible oils is a crucial challenge in food quality control, particularly due to increasing adulteration practices that compromise nutritional value and consumer trust. In this study, Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy combined with chemometric techniques were employed to authenticate commercial corn oil and detect its adulteration with lower-cost vegetable oils such as soybean and palm oils. Principal Component Analysis (PCA) was utilized for qualitative assessment and discrimination among different brands, while Principal Component Regression (PCR) and Partial Least Squares (PLS) were applied for quantitative determination of adulteration levels. Spectral analysis in the 4000–600 cm^{-1} range revealed overlapping spectral features among pure oils, making conventional differentiation challenging. However, chemometric modeling successfully distinguished pure corn oil from adulterated samples and estimated adulteration levels with high accuracy. The integration of ATR-FTIR spectroscopy with chemometric tools provides a rapid, non-destructive, and reliable approach for corn oil authentication, offering significant advantages for regulatory bodies and the food industry in ensuring product integrity.

Keywords: Corn oil; Authentication; ATR-FTIR; Principle component analysis; Principal component regression; Partial least square.

1. Introduction

Vegetable oils are typically extracted from plant sources like seeds, fruits, or vegetables, and include oils such as canola oil, soybean oil, corn oil, sunflower oil, and olive oil. Each oil has its own unique flavor, nutritional profile, and cooking properties [1-3]. Vegetable oils are an important source of dietary fat and contain a range of fatty acids, vitamins, and other nutrients that are essential for human health [4]. Vitamin E in vegetable oils acts as an antioxidant to protect the oils from oxidative damage. Vegetable oils also contain phytosterols, which have been shown to reduce cholesterol levels in the blood [5]. In addition to nutritional benefits, edible oils have economic benefits [6]. Like many other food products, edible oils adulteration is a frequent problem in food industry. The adulteration of edible oils poses a significant risk to public health, as it leads to diminished nutritional quality, increased vulnerability to oxidation, and the potential generation of harmful substances. A prevalent form of fraud in this context involves replacing costly ingredients with less expensive alternatives, resulting in the blending of high-value oils with those of lower economic worth [7,8]. Among all edible oils, olive oil is assumed to be the most expensive one and hence it is more subjected to adulteration than any other edible oil. Therefore, the majority of the published work in this task concentrate on the adulteration of olive oil with less priced oils. For example, spectroscopic methods (FT-IR, NIR, UV-VIS) have been used successfully for assessing olive oil and possible adulterant oils [9-18]. Gas chromatography was used to analyze the fatty acid composition of eight samples of edible oils with various compositions [19]. However, the current work targets the authentication of corn oil instead of olive oil. Corn oil is extracted from the seed of maize. The factors that makes corn oil popular as a cooking oil are - its low cost and neutral flavor. With only a little amount of saturated fatty acids, corn oil mostly includes polyunsaturated and monounsaturated fatty acids. The major polyunsaturated fatty acid in corn oil is linoleic acid, an essential fatty acid that must be consumed in food [20]. Currently, corn oil is vulnerable to adulteration as a result of its significant price increase on a global scale. This rise in corn oil prices can be attributed to various factors, including heightened global demand, reduced supply stemming from the COVID-19 pandemic, increased production costs, the expansion of the biofuel industry, and the recent conflict between Ukraine and Russia [21]. In this work, ATR-FTIR spectroscopy coupled with principle component analysis (PCA) will be employed for distinguishing among the most common corn oil commercial brands. The adulteration of corn oil with different ratios of less priced oils such as soy, sunflower, palm, and canola oils will be revealed. Quantitative determination of adulteration ratios will also be determined through analyzing the data with principal component regression (PCR) and partial

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least square (PLS). ATR-FTIR spectroscopy offers a non-destructive, reliable and powerful analytical approach with high efficiency and minimal sample preparation. If IR spectroscopy is coupled with chemometric tools for data analysis the analytical performance in terms of sensitivity and accuracy will be greatly enhanced and the time of analysis will also be reduced. Chemometric tools can also be directly connected to instrument software for in situ and rapid analysis for screening and automation applications. [22-24].

2. Experimental

In the current study ATR-FTIR spectroscopy was employed as a major spectroscopic technique for the assuagement of edible oils and uncovering the adulteration both qualitatively and quantitatively. Therefore, PCA was used to assess pure oil samples of different types and uncover adulterated corn oil semi- quantitatively. On the other hand, both PCR and PLS models was utilized for quantitative determination of corn oil adulteration.

2.1 Samples

PCA model: Pure oil samples: A total of 110 samples belong to five oil types and 22 commercial brands were directly purchased from the inclusive importers in Jordan. The collected edible oil samples include corn, soybean, sunflower, canola, and palm oils. The oil samples were selected based on their popularity and availability in the Jordanian market. The studied samples are listed in Table1. Adulterated (mixed) samples: to imitate adulteration of corn oil, binary mixtures of a selected brand of corn oil with soy oil and palm oil were created. Soy oil and palm oil were selected as adulterants for their minimal prices compared to corn oil. The mixing ratios were 5, 10, 15, 20, 25, 30 and 40 (v/v) %. Five mixed samples were prepared for each ratio. The total volume of each sample was 5.0 mL and the total number of adulterated samples was 70 samples. PCR and PLS models: In both PCR and PLS, the calibration set was constructed using a corn oil samples (Afia) mixed with palm oil (Yolla) and another group of samples in which palm oil was replaced with Zaiti soybeans oil. The calibration data set consisted of the following concentrations of adulterant oil (5, 8, 10, 12, 15, 20, 25, 30 and 40) %. While the validation dataset contained the concentrations (5, 10, 15, 20, 25, 35 and 40) % from the adulterant oils.

2.2 Instrumentation and data collection

ATR-FTIR spectra were collected on a Bruker Alpha spectrometer with a zinc selenide window and Deuterated Triglyceride Sulfate (DTGS) detector. The FTIR spectra were collected in the range 4000 – 600 cm^{-1} in triplicate with air as a background; the resolution was 4 cm^{-1}.

2.3 Data analysis and software

For data processing and analysis, MATLAB 7.0.4 MathWorks, MA, USA and PLS_Toolbox 4.0 Eigenvector Research, Inc., WA, USA were employed.

Results and Discussion

The ability to identify different edible oil types through their FTIR spectra is a very important task in order to uncover any possible oil adulteration. At first a visual inspection of the obtained oil spectra has been carried out in attempt to discriminate among the different pure oils used in this study. Therefore, the IR spectra of corn, sunflower, canola, soybean and palm oils in the range 4000-600 cm^{-1} are displayed in Figure 1, in order to identify characteristic bands in each oil.

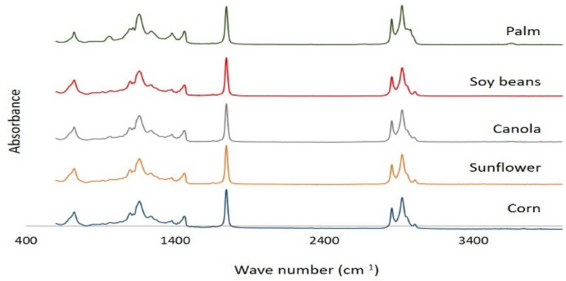

Figure 1: FTIR spectra of corn (Afia), sunflower (Golden), canola (Olite), soybean (Al-amer) and palm (Yolla) oils.

In this figure, one commercial brand was selected from each oil type for the purpose of initial visual investigation. As it appears in Figure 1, all oil samples reveal almost identical spectral features. The bands in the range 2850-2960 cm^{-1} are attributed to symmetric and asymmetric stretching vibrations of the C-H bond in CH_2 and CH_3. The band 1700-1790 cm^{-1} is attributed to carbonyl (C=O) group. The bending vibrations of CH_2 and CH_3 groups appear in the range 1020-1500cm^{-1}. The band at 722 cm^{-1} belongs also to hydrocarbons. The high similarity of the obtained spectra and the absence of characteristic bands makes it very difficult even for IR experts to distinguish among these spectra and hence, assessment of these oils in this

range of IR region would be a very challenging task. The IR spectra of corn oil mixed samples also revealed no special visual characteristics than the pure oil spectra (figure is not shown). Hence, uncovering adulterated corn oil through classical spectral analysis protocol is almost impossible. On the other hand, the ability of FT-IR to distinguish among the different brands of a given oil has been examined. The most two expensive oils in this study (corn and sunflower) were selected and examined. Five samples of each corn and sunflower oil brand that appear in Table 1 were studied. This means that 30 samples of corn oil of six brands, and similar number of sunflower oil brands were investigated. Figure 2, represents one single spectrum of each corn oil brand (Al-Amer, Zaiti, Durra, Afia, Karam zamzam and Golden corn). Figure 3, represents the FT-IR spectra of one single spectrum of each sunflower oil brands (Al-Amer, Karam zamzam, Zaiti, Shaban, Sunny and Golden). In Figures 3 and 4, very limited or no differences among the spectra of the commercial brands were detected. As a result, discriminating between the different corn oil brands or that of sunflower was not possible.

3.1 PCA for assessment of pure oils

The selected oils in this study belong to five main types, those are corn, sunflower, soybean, canola and palm oils. Five samples of each oil in Table 1 were FT-IR scanned and recorded. Therefore, the number of studied samples were as follows: 30 samples of each corn and sunflower oils, 20 samples of each of soybean and canola oils and 10 samples of palm oil. So, the total number of samples was 110. All samples were scanned in a similar manner; each sample is represented by 1666 absorption data points. Therefore, for the purpose of PCA application, a single data matrix that include all the 110 samples was constructed. The dimensions of the matrix were 110 columns x 1666 rows. The first few PCs were calculated, and displayed in Table 2. PCA has the ability of analyzing entire data set simultaneously and find the similarities and differences among columns (objects or samples).

Table 1: Types and brands of oil samples used in this study

#	Oil type	Brand	Number of samples
1	Corn	Al-Amer	5
2	Corn	Zaiti	5
3	Corn	Durra	5
4	Corn	Afia	5
5	Corn	Karam zamzam	5
6	Corn	Golden Corn	5
7	Soybean	Al-Amer	5
8	Soybean	Addyafe	5
9	Soybean	Sun top	5
10	Soybean	Zaiti	5
11	Sunflower	Karam zamzam	5
12	Sunflower	Al-Amer	5
13	Sunflower	Zaiti	5
14	Sunflower	Shaban	5
15	Sunflower	Sunny	5
16	Sunflower	Golden	5
17	Canola	Olite	5
18	Canola	Afia	5
19	Canola	Noor	5
20	Canola	Newland	5
21	Palm	Yolla	5
22	Palm	Tala	5

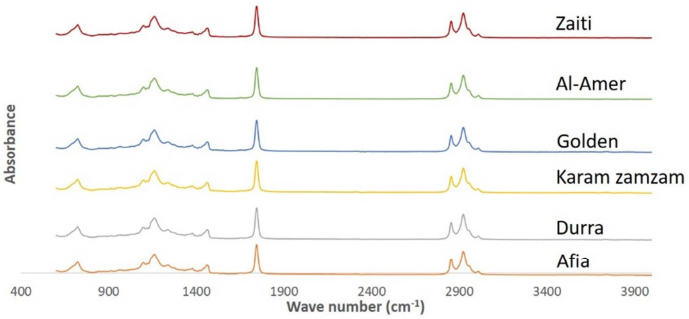

Figure 2: FTIR spectra of corn oil brands.

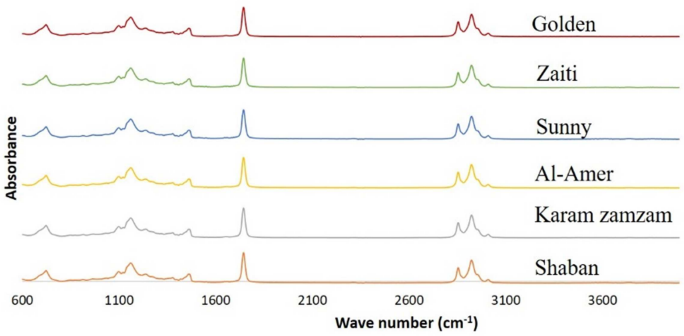

Figure 3: FTIR spectra of sunflower oil brands.

Therefore, PCA is considered a size reduction algorithm without losing significant information.
The best PCA score plot was obtained using the first two PCs that express about 97% of the total variation in the above dataset. The obtained PCA score plot is shown in Figure (4).

Table 2: Percent variance captured by PCA model application to the FT-IR spectral data of all pure oils in this study

Principal Component	Eigenvalue of (Cov(X))	% Variance Captured (This PC)	% Variance Captured Total
1	5.51E-02	77.5	77.5
2	1.38E-02	19.41	96.91
3	1.36E-03	1.92	98.82
4	5.03E-04	0.71	99.53
5	1.41E-04	0.2	99.73

PCA was applied to the data matrix was constructed using full FT-IR spectra of corn, sunflower, soy bean, canola and palm oils.

In PCA, spectra of similar samples or samples that have identical spectra tend to form what is so called a cluster. In a cluster, points are usually very close to each other and a way from other points in the plot. Each point in the PCA scores plot represents a full FT-IR spectrum. The more differences in the spectra the higher the distances between their points in the PCA plot. In the current PCA scores plot (Figure 4), five independent clusters can be recognized. Those are an independent cluster for each of corn oil, sunflower, soybeans, canola and palm. In the PCA score plot, the first PC that expresses more than 77% of the total variation in the dataset was able to distinguish all types of oils except that it failed in distinguishing among corn

and sunflower oil samples. This means that the PCA algorithm was able to find unique features in the spectra and use it to discriminate among all spectra. Also, this PC1 could not capture very unique features among corn and sunflower oil samples due to the high similarity in their spectra. Figures 5 and 6 show the resulted PCA scores plot using PC1 and PC2, of all corn oil brands and all corn and sunflower brands in this study, respectively. The data matrix in each case was constructed in a similar way to the previous data matrices that were described formerly in this study. Once again, despite the great similarities in the IR spectra of all brands, PCA was successful in assessing each brand in both cases (corn and sunflower oils). Each commercial brand of the two oils formed a well resolved cluster in the PCA model except in case of Shaban and Al-Amer sunflower oils that were overlapping between the clusters of the two oils as was detected in Figure 6. The failure in distinguishing these two brands of sunflower oil reflects their greater spectral similarities.

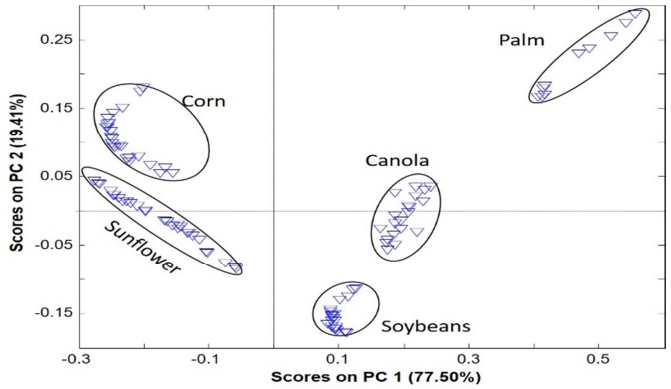

Figure 4: PCA scores plot of all pure oils in this study

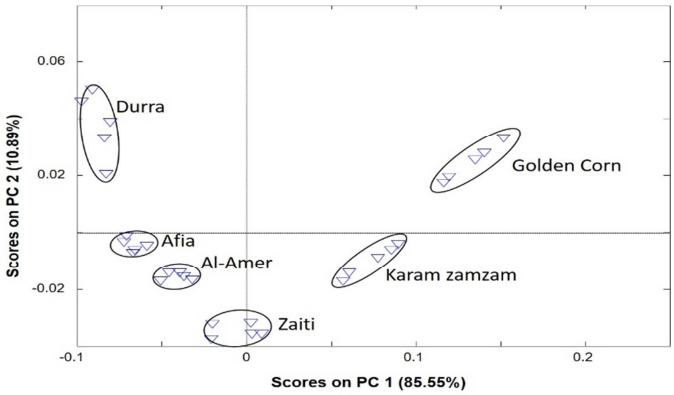

Figure 5: PCA scores plot of all commercial brands of corn oil in this study

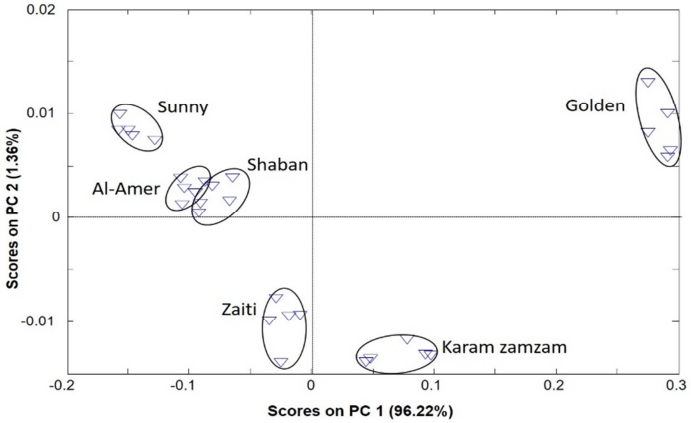

Figure 6: PCA scores plot of all commercial brands of sunflower oil in this study.

3.2 ATR-FTIR and PCA to uncover corn oil adulteration

The adulterated corn oil samples are imitated through mixing the corn oil with less expensive oils with different mixing ratios. The mixed corn samples in this study were binary mixtures of corn from one side, palm and soybean oils in the other side (Table 3). To avoid crowdedness of the PCA model and to achieve best PCA vision, two new PCA scores calibration plots were constructed. One model contains only pure samples of the selected corn and soybean oils and the other one involves again the selected pure samples of corn and palm oil samples. These figures were used as a calibration models to uncover adulteration of corn oil with different ratios of soy beans and palm oils, Figures 7 and 8, respectively The first few PCs were calculated in each case. Tables 4 and 5 show the corresponding captured variance in the dataset of each calculated PC. The best score plots were obtained using the first two PCs in corn-soybeans oils case were used and expressed more than 99% of the variations in the data set. And in case of corn-palm oils, the best PCA plot involved the first and the third PCs (more than 83% of the total variation were expressed). After having two calibration PCA plots, spectra of all adulterated samples of all ratios were recorded. The spectra of adulterated samples using the soybeans oil were organized in a single data matrix and another data matrix was constructed using the spectra from adulterated samples with palm oil. Figure 7, shows the resulted PCA plot after applying the reconstructed PCA calibration model of pure corn and soybeans oils to the dataset of adulterated samples of corn oil with different ratios of soybeans oil (5,10, 15, 20, 25, 30 and 40) %.

Table 3: Composition of oil mixtures (v/v) % used for PCA application

Corn oil (Afia), mL	Palm oil (Yolla) or Soybean oil (Zaiti), mL	%Palm oil or Soybean oil
4.75	0.25	5
4.5	0.5	10
4.25	0.75	15
4	1	20
3.75	1.25	25
3.5	1.5	30
3	2	40

Total sample volume is 5.0 mL (each mixture was prepared in 5 replicates, n=70).

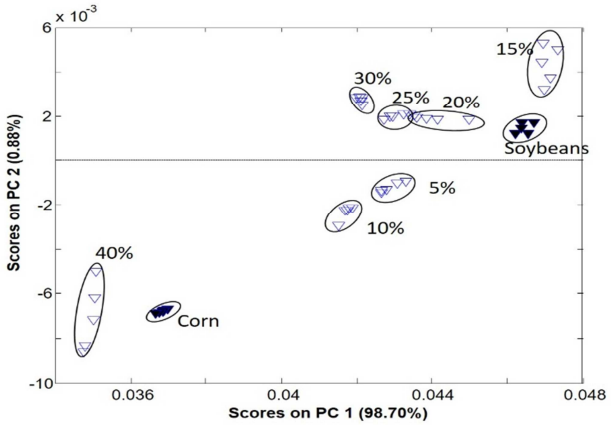

Figure 7: The results of applying spectra of corn adulterated samples with soybeans oil (empty triangles) to calibration PCA scores plot of pure corn and soybeans oil samples (solid triangles), percentages represent the soybean oil ratios

As can be seen in this figure successful detection of all adulterated (mixed) oil samples was achieved successfully. Not only that but also each adulteration ratio formed almost a separated cluster which reflects the ability of PCA for detecting the mixing ratio for a certain limit. On the other hand, Figure 8 represents the result of applying the pre-constructed PCA model of pure corn and palm oil samples to the data matrix than contains the spectra of all adulterated samples with the specific adulteration ratios. The PCA showed excellent results in detecting these adulterated samples through forming well resolved clusters in the PCA model away from the clusters of pure samples of corn and palm. Each one of these clusters represents samples of a specific mixing ratio. The resulting PCA plots in Figures 7 and 8, may also be used for determining adulteration ratio for future samples. However, exact adulteration ratio may not be accurate due to fact that all possible mixing ratios must be included in the model with is very difficult to apply. Another reason is that in case if the test sample located between two

clusters then the exact determination of mixing ratio becomes very difficult relying on the PCA model alone especially that many clusters in the above PCA models are very close to each other. Therefore, another analysis tool must be employed to try to determine exact mixing ration with good certainty. Hence, PCR and PLS were employed for the purpose of determining the mixing ratio accurately.

Table 4: Percent variance captured by PCA model applied to FT-IR spectral data of corn and soy beans oils and their binary mixtures.

Principal Component	Eigenvalue of (Cov(X))	% Variance Captured (This PC)	% Variance Captured Total
1	1.83E-03	98.70%	98.70%
2	1.63E-05	0.88%	99.58%
3	7.51E-06	0.40%	99.98%
4	1.50E-07	0.01%	99.99%
5	5.79E-08	0.00%	99.99%

Table 5: Percent variance captured by PCA model applied to FT-IR spectral data of corn and palm oils and their binary mixtures.

Principal Component	Eigenvalue of (Cov(X))	% Variance Captured (This PC)	% Variance Captured Total
1	2.50E-01	81.88%	81.88%
2	5.09E-02	16.64%	98.52%
3	4.39E-03	1.44%	99.95%
4	5.08E-05	0.02%	99.97%
5	3.61E-05	0.01%	99.98%

3.3 PCR and PLS for quantitative determination of corn oil adulteration

In both PCR and PLS analysis a calibration set was constructed using a selected adulteration ratio from Table 3. So, corn oil samples were mixed with a selected brand of palm oil (Yolla) and another group of samples, where palm oil was replaced with Zaiti soybeans oil. The calibration data set consisted of the following concentrations of adulterant oil (5, 8, 10, 12, 15, 20, 25, 30 and 40) %. While the validation dataset contained the concentrations (5, 10, 15, 20, 25, 40 and 100) % from the adulterant oils for the construction of PCR and PLS chemometric calibrations.

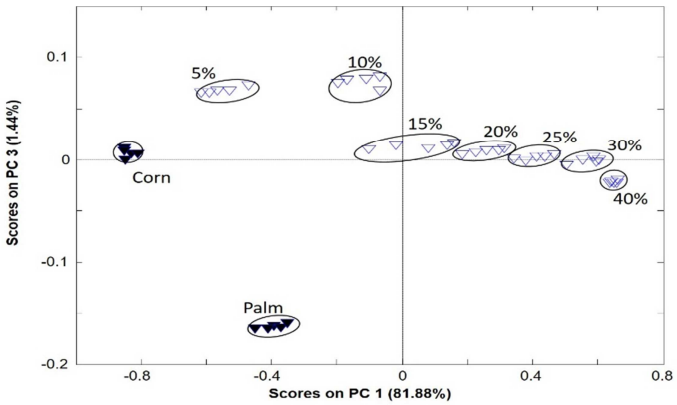

Figure 8: The results of applying spectra of corn adulterated samples with soybean oil (empty triangles) to calibration PCA scores plot of pure corn and palm oil samples (solid triangles), percentages represent the palm oil ratio.

The calibration PCR and PLS models were constructed after auto-scaling as a preprocessing step, cross validation method was applied through the leave-one-out procedure. In order to determine the number of components for each model the RMSEC (root mean square error of calibration) values were calculated for the first few components using equation (1) [25]. The optimum number of components was determined in a way that additional components can't be count unless they improve the RMSEC by at least 2%, in addition to a careful visual investigation of the constructed models. As a result, five components were used in case of the PLS model while four components were employed for the PCR model.

$$\boldsymbol{Press} = \sum_{i=1}^{n}(\hat{ci} - ci)^2 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where ci is the reference concentration for the i sample and $\hat{ci}$ represents the estimated concentration. Plots of the measured versus predicted values of the palm oil ratio using the PCR and PLS calibration and validation sets were created. The obtained equations, R^2, percentage recoveries and relative were calculated and indicated in Table 6. The average recovery value for PCR model was 100.07% for calibration and 97.71% for validation set. In case of the PLS model, 98.92% and 97.75% recoveries were obtained for both calibration and validation sets respectively. On the other hand, applying both PCR and PLS on the corn-soybeans mixed samples revealed an average recovery values of 94.45% and 89.48% for PCR calibration and validation set respectively, and in case of PLS application the corresponding recovery values were 95.02% and 91.92 for calibration and validation sets, respectively (Table 7). The linearity of all the above PCR and PLS models in addition to the slope of the obtained curves are summarized in Table 8. From these numerical data (Tables 6,7 and 8), the recovery values were found very satisfactory for the validity of both PCR and PLS models. Also both PCR and PLS models exhibited very comparable results in terms of analytical chemistry. The obtained results are in good agreement with those obtained upon using FT-NIR spectroscopy and chemometrics [9, 26].

Table 6: The results of applying both PCR and PLS to the adulterated corn oil samples with different ratios of palm oil

PCR					
Calibration			**Validation**		
Actual %	% Predicted	%recovery	Actual %	% Predicted	%recovery
5	5.00	100.96	5	3.71	74.96
8	7.90	98.71	10	9.71	97.00
10	10.10	100.59	15	17.38	116.17
12	12.00	99.92	20	21.44	107.17
15	15.00	100.14	25	25.01	99.82
20	20.00	99.85	40	39.60	99.07
25	25.10	100.30	100	90.58	90.64
30	29.90	99.72			
40	40.00	100.07			
	Average recovery	100.03		Average recovery	97.71
PLS					
Calibration			**Validation**		

Actual %	% Predicted	%recovery	Actual %	% Predicted	%recovery
5	4.86	97.20	5	3.80	76.00
8	8.24	103.00	10	9.81	98.10
10	10.53	105.30	15	17.20	114.67
12	11.32	94.33	20	19.31	96.55
15	15.08	100.53	25	25.23	100.92
20	18.18	90.90	40	39.22	98.05
25	23.76	95.04	100	91.48	91.48
30	32.11	107.03			
40	38.77	96.93			
	Average recovery	98.92		Average recovery	97.75

Table 7: The results of applying both PCR and PLS to the adulterated corn oil samples with different ratios of soybean oil

PCR					
Calibration			**Validation**		
Actual %	% Predicted	%recovery	Actual %	% Predicted	%recovery
5	5.05	101	5	4.12	82.96
8	7.32	91.5	10	9.01	88.24
10	9.56	95.6	15	13.83	91.17
12	11.12	92.667	20	19.09	95.46
15	14.32	95.467	25	22.71	90.82
20	18.01	90.05	35	31.17	89.07
25	25.11	100.44	40	36.26	88.64
30	27.22	90.733			
40	37.04	92.6			
	Average recovery	94.45		Average recovery	89.48
PLS					
Calibration			**Validation**		

Actual %	% Predicted	%recovery	Actual %	% Predicted	%recovery
5	4.74	94.88	5	3.52	70.4
8	7.69	96.09	10	12.05	120.5
10	9.30	93.01	15	13.55	90.333
12	10.79	89.94	20	16.88	84.4
15	13.76	91.73	25	26.9	107.6
20	18.93	94.63	35	29.46	84.171
25	24.33	97.32	40	34.41	86.025
30	28.16	93.86			
40	38.01	95.02			
	Average recovery	94.05		Average recovery	91.92

Table 8: The Slope and R2 value obtained upon PCR and PLS application to the adulterated (mixed) corn oil samples

		PCR		PLS	
Oil mixture		Calibration	Validation	Calibration	Validation
Corn-Palm	Slope	0.998	0.88	1.000	0.89
	R^2	1.000	0.998	1.000	0.995
		Calibration	Validation	Calibration	Validation
Corn-Soybeans	Slope	0.913	0.811	0.904	0.784
	R^2	0.956	0.901	0.911	0.921

3. Conclusions

In this work an attempt to assess different types of cocking oils including (corn, sunflower, soybeans, canola and palm) has been achieved successfully using ATR-FTIR spectroscopy coupled with PCA for data analysis. The IR spectra were collected for all samples in the range between 4000 – 600 cm^{-1}. Six common commercial brands from each of corn and sunflower oils were also obtained and IR scanned. PCA was also employed for analyzing the recorded spectra. Once again, this technique was successful in distinguishing among the different commercial brands of each oil.

In the second part of this work, corn oil was selected as a potential target for adulteration through mixing it with different ratios of less expensive oils (palm and soybeans). Uncovering adulterated samples using a PCA scores plot was also achieved successfully. However, quantitative determination of adulterating ratios in both cases can't be achieved accurately. Therefore, only semi-quantitative determination was done using both FTIR and PCA. For quantitative determination of corn oil adulteration, PCR and PLS were employed for data analysis. Successful and a very convenient determination of adulteration was achieved in the case mixing the corn oil with multiple ratios with palm oil and a for a less extent when soybeans oil used for adulteration. Greater number of cocking oil types and brands may also be used to expand the study and build a data base of IR spectra of cocking oils in the Jordanian market for more advanced analysis.

Conflicts of interest

There are no conflicts to declare.

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