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RESEARCH ARTICLE

Identification of adulterants (sunflower oil and soybean oil) in grapeseed oil (*Vitis vinifera* L.) and chia oil (*Salvia hispanica* L.) by FT-MIR spectroscopy and chemometric

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ABSTRACT

Grapeseed and chia oils are important for their fatty acids profile. Both oils are considered gourmet and due their high retail prices they are susceptible to adulteration. FT-MIR was used with chemometric analysis to identify and quantify the adulterants (sunflower and soybean oils) in grapeseed and chia oils. A ternary mixture system was designed for each oil using as adulterants sunflower and soybean oils (2-50%, w/w). Soft independent modelling by class analogy (SIMCA) discriminated adulterated samples and pure samples (100% of recognition and rejection). Partial least squares (PLS) showed the best calibration result (R^2c: ≥ 0.95; SEC: 2.22-2.87) and validation (R^2v: ≥ 0.99; SEP: 2.25-3.13). The models could be applied to identify and quantify two adulterants: sunflower and soybean oils in grapeseed and chia oils.

Keywords: Adulteration; Chemometric analysis; Chia oil; FT-MIR spectroscopy; Grapeseed oil; Ternary mixture

INTRODUCTION

Grapeseed (*Vitis vinifera* L.) and chia (*Salvia hispanica* L.) oils are an important source of polyunsaturated fatty acids such as linoleic acid (omega-6) and linolenic acid (omega-3). Both fatty acids are indispensable for human beings because they are beneficial in reducing total cholesterol (Kulczyński et al., 2019). Thus, grapeseed and chia oils are considered gourmet, their high retail prices might promote adulteration. Adulteration of these gourmet oils is important because of safety, quality, and economical reasons (Everstine et al., 2013).

Adulteration in edible vegetable oils may be achieved by diluting the oil with one (binary mixture) or two (ternary mixture) lower costs and lower quality oils. Some examples of oils commonly used for adulteration are sunflower, soybean, corn, palm, peanut, and sesame (Gorkem et al., 2017), these oils have been used as adulterants, due their low retail cost and their availability (Azadmard-DamirchI and Torbati, 2015).

Adulteration of vegetable oils practices have evolved rapidly. Thus, their detection is increasingly difficult. Several techniques have been developed for the analysis of adulterants in edible vegetable oils, for example: high-performance liquid chromatography (HPLC) (De la Mata-Espinosa et al., 2011), nuclear magnetic resonance (RMN) (Zhang et al., 2013), gas chromatography (GC) (Ruiz-Samblás et al., 2012), fluorescence spectroscopy (Ge et al., 2014; Mburu et al., 2021), near infrared spectroscopy (Mburu et al., 2021), Raman spectroscopy (Mburu et al., 2021; Farley et al., 2016) and Mid-Infrared spectroscopy (Jiménez-Sotelo et al., 2016).

Fourier Transform Mid-Infrared spectroscopy (FT-MIR) with chemometric analysis is rapid, eliminates the use

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of solvents and reagents and it does not require sample preparation. FT-MIR coupled with chemometric analysis has been used to identify the adulterants of various edible vegetable oils such as avocado, olive, coconut, and canola (Ozren, 2016) among others.

Akin et al. (2019) and Rodríguez et al. (2019) reported respectively grapeseed and chia oil adulteration using FT-MIR combined with chemometrics, however, these studies are focused on a binary adulteration system. This work reports for first time the use of FT-MIR and chemometric analysis to quantify adulterants (sunflower oil and soybean oil) in grapeseed and chia oils based on a ternary mixtures system.

MATERIALS AND METHODS

Chemicals

Fatty acid methyl esters (FAMEs) were acquired from Restek Corp. (Bellefonte, PA, USA).

Samples

Twenty samples (five of each) of grapeseed, chia, sunflower, and soybean oils were purchased from shops and supermarket in Mexico City, México. Oil brands purchased are the most consumed. All oils were stored in the dark at room temperature until analysis.

Methods

To confirm the quality of the oils used (grapeseed, chia, sunflower, soybean), the peroxide values and iodine values were carried out according to the AOAC (2002). Chemical analyses were carried out in triplicate.

Fatty acid analysis

In order to confirm the authenticity of the oils used, their fatty acid composition was determined by GC. GC was used to analyze the Fatty Acid composition (FA) of the oils used (grapeseed, chia, sunflower, soybean oils). FAMEs were prepared according to IUPAC method (IUPAC, 1979) and the analysis was according to Jiménez-Sotelo et al. (2016). Fatty acid composition was made using a gas chromatograph Clarus 500 (Perkin Elmer, Massachusetts, USA) equipped with a flame ionization detector (FID). A The analysis was carried out with a SP-2380 column (100 m length, 0.25 mm i.d., 0.20 μm film thickness, Supelco Inc. Bellefonte, PA, USA). Each sample was analyzed in triplicate and averaged. The Fatty Acid Methyl Esters were identified using the retention time. The conversion of Fatty Acid Methyl Esters to Fatty Acids was carried out according to the AOAC (2002). The results were expressed as the percentage of total fatty acids based on peak area.

Selection of a representative sample for each type of oil

The COMPARE algorithm (Spectrum, version 10.5, PerkinElmer, Massachusetts, USA) was used to compare the FT-MIR spectra of the twenty brands (five of each) of grapeseed, chia, sunflower, and soybean oils and thus select a representative sample for each type of oil.

For example, for grapeseed oil (Table 1), brand A was chosen since this brand (A) had the highest correlation coefficient (0.9998) compared to the others brands (B, C, D, E). Thus, brand A has the most representative spectral features unlike brands B, C, D and E. Selection of chia, sunflower and soy oil brands was made by the same procedure.

Preparation of adulterated ternary mixtures

Grapeseed and chia oil were adulterated with sunflower and soybean oil in ternary mixtures. A mixture design (Minitab Statistical Software, version 18.1, State College, PA, USA) was made to randomly select 70 adulterated ternary samples of each oil. Preparation of adulterated ternary mixtures was from 2 to 50%. The calibration data included 47 samples, whilst validation data included 23 samples. This percentage of adulteration (2-50%, w/w) was selected according to previous studies (Quiñones-Islas et al., 2013; Jiménez-Sotelo et al., 2016).

FT-MIR spectra acquisition

FT-MIR spectra of pure oils (grapeseed, chia, sunflower, and soybean) and adulterated samples were acquired using a Spectrophotometer Fourier transform infrared (Frontier, Perkin Elmer, Massachusetts, USA) equipped with a deuterated triglycine sulphate detector. The sampling station was equipped with an overhead attenuated total reflection accessory (ATR) with a detachable ZnSe crystal. The ZnSe ATR crystal surface was a 45° parallelogram with mirrored angled faces, with ten nominal internal reflections. The FT-MIR spectra were scanned over the interval of 4000-550 cm^{-1}, averaging 64 scans, resolution of 4 cm^{-1} and in absorbance units (A). The FT-MIR spectra of the samples were collected against a background of air. The results

Table 1: Coefficients between the brands of oils and their blends.

Brand	Grapeseed oil	Chia oil	Sunflower oil	Soybean oil
	Correlation coefficient[a]	Correlation coefficient[a]	Correlation coefficient[a]	Correlation coefficient[a]
A	0.9998[b]	0.9981	0.9997[b]	0.9957
B	0.9970	0.9975	0.9991	0.9986
C	0.9986	0.9989[b]	0.9989	0.9973
D	0.9942	0.9972	0.9985	0.9991[b]
E	0.9993	0.9964	0.9976	0.9964

[a]Match between the FT-MIR absorbance of the oil and the blend (coefficient must be 1.000). [b]Oil brand selected to develop the models.

were recorded in triplicate using the Spectrum software version 3.01.00 (PerkinElmer, Massachusetts, USA).

Chemometric analysis

Identification with SIMCA (Soft Independent Modelling by Class Analogy)

The models to identify among pure oils (grapeseed, chia, sunflower, soybean) and adulterated ternary mixtures were developed with the software AssureID version 10.4 (PerkinElmer, Massachusetts, USA).

Two SIMCA models were constructed (one of grapeseed oil and one of chia oil). In each SIMCA model two classes were created: 1) grapeseed or chia oil; 2) sunflower oil; 3) soybean oil and 4) adulterated ternary mixtures. The SIMCA models were calibrated using 47 spectra from each class, and the validation set was developed with 23 FT-MIR spectra from each class.

The models optimization consisted in using spectral pre-treatments: spectral range (620-550 cm^{-1}), remove ambient filters such as H_2O and CO_2, normalization such as multiplicative scatter correction (MSC), smoothing Savitzky-Golay filter (window of 9 and 13 points) baseline correction type Offset.

The performance of SIMCA models were evaluated through: 1) Principal components (PC) projection and indicates separated classes; 2) the interclass distance (which must be over three and indicates the similarity among classes); 3) the percentages of recognition and rejection (both must be 100%). SIMCA models were submitted to an external validation. Total distance and residual distance were evaluated (PerkinElmer, 2014a).

Quantitative models

To develop the models to quantify adulteration of grapeseed and chia oils, the software Spectrum QUANT+ version 10.4 (PerkinElmer, Massachusetts, USA) was used. The software QUANT+ counts with Partial Least Square (PLS) and Principal Components Regression (PCR) algorithms, which correlates spectral data and percentage of adulteration. The calibration set included 47 adulterated ternary samples, and the validation set included 23 adulterated ternary samples.

The models optimization consisted in using spectral pre-treatments: normalization (multiplicative scatter correction, MSC) and baseline correction (First derivative, 5 points). Performance of the models was evaluated through 1) factors (latent variables); 2) coefficient of determination (R^2c, must be close to 1); 3) Standard Error of Calibration (SEC, must be lowest) (PerkinElmer, 2014b).

Quantitative models were submitted to an external validation. Coefficient of determination (R^2v, which must be as close to 1 as possible) and Standard Error of Prediction (SEP, must be as low as possible) were evaluated (PerkinElmer, 2014b).

RESULTS AND DISCUSSION

Chemical analysis

The peroxide values ranged from 0.39 to 0.79 meq/kg, and the iodine values were 107.08 to 197.73 cgI_2/g for all oils (Table 2). The free fatty acids (%) were among 0.01 and 0.02 for all oils. The physicochemical parameters (peroxide values, iodine values and free fatty acids) of the oils (grapeseed, chia, sunflower, and soybean oils) were within the quality limits established by official methods (DGN, 2011a; DGN, 2011b; Codex, 2013; DGN, 2013; DGN, 2017).

Fatty Acid profile

The main Fatty Acid in grapeseed and soybean oils is linoleic (C18:2) with 66.59% and 53.54% respectively. In chia oil, the main Fatty Acid was linolenic acid (C18:3) with 59.18%. In sunflower oil the main fatty acid was oleic acid (C18:1) with 51.75% (Table 2). Regarding the results, grapeseed oil, chia oil, sunflower oil and soybean oil presented fatty acids composition in agreement with legislation (Codex, 2013; DGN, 2011a; DGN, 2011b; DGN, 2013; DGN, 2017).

FT-MIR spectra

Fig. 1a depicts FT-MIR spectra of: pure grapeseed, sunflower and soybean oils, whilst Fig. 1b presents FT-MIR spectra of: pure chia, sunflower and soybean oils. The first

Table 2: Peroxide values (meq/kg), iodine values (cgI_2/g), Free Fatty Acids (%) and Fatty Acid (%) of grapeseed oil, chia oil, sunflower oil and soybean oil.

	Grapeseed oil	Chia oil	Sunflower oil	Soybean oil
Peroxide (meq/kg)	0.39±0.01 *(≤ 2)*[a]	0.79±0.01 *(≤ 2)*[a]	0.39±0.01 *(≤ 2)*[b]	0.39±0.01 *(≤ 2)*[c]
Iodine (cgI_2/g)	140.42±0.40 *(128-150)*[d]	197.73±0.33 *(191-199)*[e]	107.08±0.06 *(75-115)*[b]	132.67±0.21 *(118-139)*[c]
Free Fatty Acids (%)	0.01±0.05 *(≤ 0.05)*[a]	0.01±0.17 *(≤ 0.1)*[e]	0.02±0.29 *(≤ 0.05)*[b]	0.02±0.13 *(≤ 0.05)*[c]
Fatty Acids (%)				
C14:0	0.04±0.46	0.05±0.48	n.d.	n.d.
C16:0	7.16±0.07	7.92±0.08	5.05±0.11	10.85±0.28
C18:0	4.39±0.08	3.87±0.13	4.66±0.10	4.45±0.11
C18:1	21.21±0.29	4.56±0.57	51.75±0.09	24.52±0.64
C18:2	66.59±0.63	23.86±0.64	37.98±0.80	53.54±0.39
C18:3	0.37±0.05	59.18±0.12	0.42±0.01	6.54±0.17
C20:0	0.23±0.38	n.d.	0.21±0.02	0.01±0.01

Numbers in italics and parentheses correspond to data reported in the literature ([a]NMX-F-223-SCFI-2011, [b]NMX-F-050-SCFI-2013, [c]NMX-F-252-SCFI-2011, [d]CODEX, 2013, e NMX-F-592-SCFI-2017). n.d., not detected

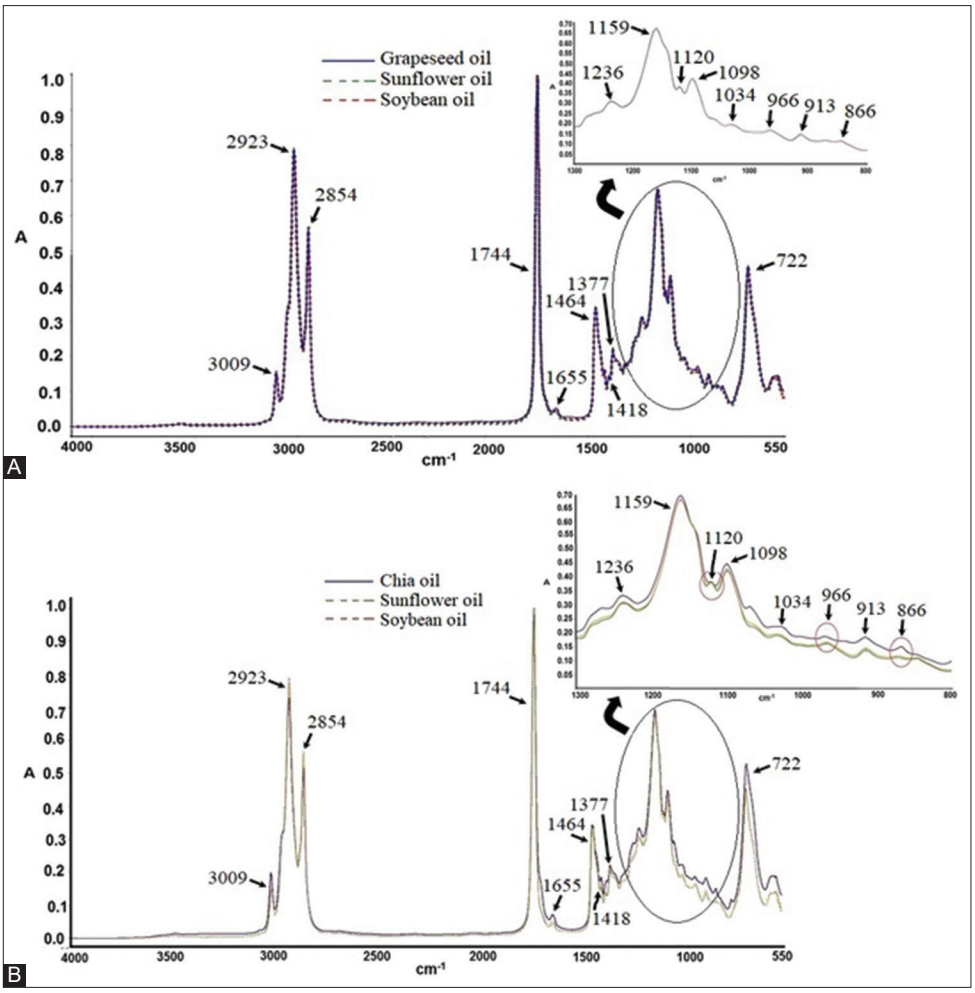

Fig 1. FT-MIR spectra of : a) grapeseed oil, sunflower oil and soybean oil; b) chia oil, sunflower oil and soybean oil.

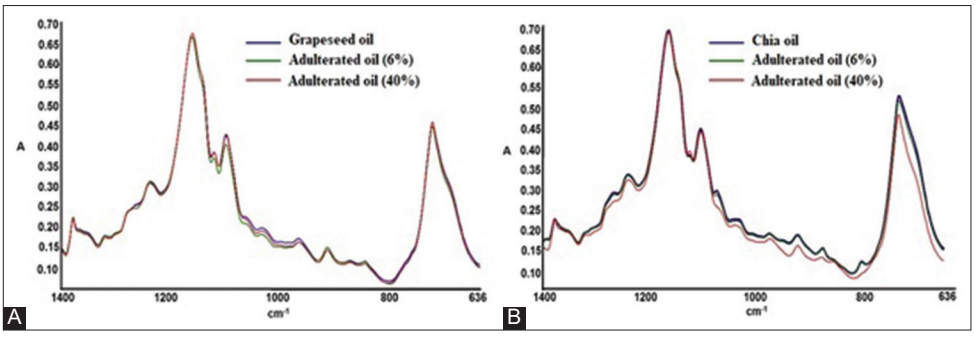

Fig 2. FT-MIR spectra in the fingerprint region (1400-600 cm^{-1}) of: a) grapeseed oil adulterated: b) chia oil adulterated.

major band at 3009 cm^{-1} is due the stretching of *cis* C=C (Vlachos et al., 2006) this band indicates the degree of triglyceride unsaturation (Rohman, 2017). At 2923 cm^{-1} and 2854 cm^{-1} is due the aliphatic group CH_2. The region at 1744 cm^{-1} is due C=O of carboxylic acids, ketones and aldehydes. The region at 1655 cm^{-1} is due the stretching of *cis* bond C=C from disubstituted olefins. The band at 1464 cm^{-1} is due the aliphatic groups CH_2 y CH_3. The band at 1418 cm^{-1} is due C-H bonds from *cis*-disubstituted olefins, whereas at 1377 cm^{-1} is due the CH_3 group.

The region of 1236 cm^{-1}, 1159 cm^{-1}, 1120 cm^{-1}, 1098 cm^{-1} and 1034 cm^{-1} are due C-O stretching vibrations from ester groups in triglycerides (Rosas-Mendoza et al., 2017). The peaks at 966 cm^{-1} and 913 cm^{-1} are due *trans* double bonds (C=C) and *cis* double bonds (C=C) (Jović et al., 2013). The peak at 866 cm^{-1} is attributed to the OH group (Yacob and Mohmedahmed, 2017). Finally, at 722 cm^{-1} is assigned to the CH_2 from *cis*-disubstituted olefins (Guillen and Cabo, 1997). The band assignation is in agreement with Guillen and Cabo (1997) and Jiménez-Sotelo et al. (2016).

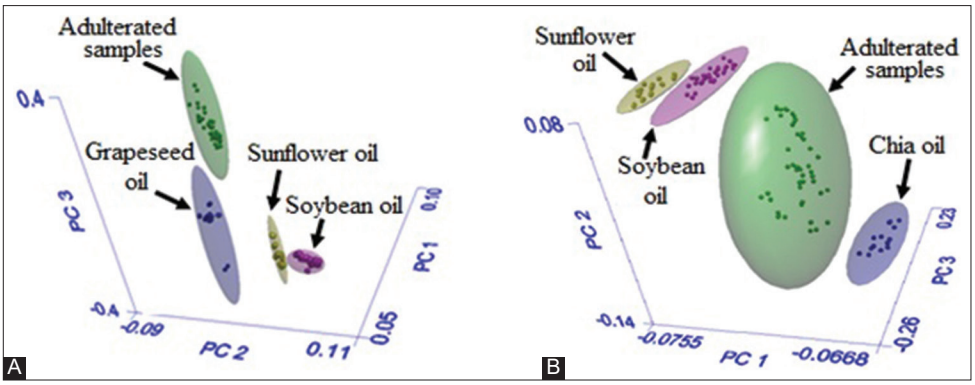

Fig 3. Three- dimensional principal component analysis scores plot of the populations derived from SIMCA: a) grapeseed oil, sunflower oil, soybean oil and adulterated samples; b) chia oil, sunflower oil, soybean oil and adulterated samples.

Fig. 2 (a, b) shows FT-MIR spectra in the fingerprint region (1400-600 cm^{-1}) of adulterated grapeseed and chia oils respectively. FT-MIR spectra from pure oils (grapeseed and chia) and adulterated samples presented a correlation among spectral changes and percentages of adulteration of each sample. The above-mentioned is important for the development of predictive models.

Multivariate analysis

Discrimination by SIMCA

Fig. 3(a,b) shows the spatial distribution by SIMCA. The SIMCA models properly discriminated among pure grapeseed oil, adulterant oils (sunflower and soybean) and adulterated ternary samples (Fig. 3a). Also, SIMCA models properly identified among pure chia oil, adulterant oils (sunflower and soybean) and adulterated ternary samples (Fig. 3b). The hyperboxes represents a 99% confidence limit (PerkinElmer, 2014a).

This spatial distribution is reflected in the interclass distance (ID). An ID higher than 3 indicates classes are separated, and therefore they are different (PerkinElmer, 2014a). Therefore, a higher interclass distance indicates, a better separation between the classes. The interclass distance between the classes of grapeseed oil and adulterated oil was 13.2, whilst interclass distance between the classes of chia oil and adulterated oil was 54.9.

Table 3 shows the percentages of recognition (sensitivity) and percentages of rejection (specificity) of the classes derived from SIMCA models. SIMCA models showed recognition and rejection rates of 100% for all classes. Percentages of recognition indicates the percentage of samples (spectra) belonging to the class being analyzed that are correctly identified by the SIMCA model, whilst the percentage of rejection represents the percentage of samples (spectra) not belonging to the class that is being analyzed (PerkinElmer, 2014a). For example, if the class that is being analyzed is grapeseed oil, 47 spectra belong to this class and therefore all were recognized as grapeseed oil, giving 100% of recognition (47 out of 47). One hundred forty-one spectra do not belong to the grapeseed oil class were rejected, giving 100% of rejection (141 out of 141). The same happened to the classes defined as sunflower oil, soybean oil and adulterated samples: 100% of recognition and 100% of rejection (Table 3). The same happened for the SIMCA model of chia, sunflower, soybean oils, and adulterated ternary samples. The above indicates that the SIMCA models correctly classified all samples with a high confidence limit (99%).

Table 3. Percentages of recognition and rejection between the classes of grapeseed, sunflower, soybean oils, and adulterated ternary samples.

Class	Recognition (%)	Rejection (%)
Grapeseed oil	100 (47/47)	100 (141/141)
Sunflower oil	100 (47/47)	100 (141/141)
Soybean oil	100 (47/47)	100 (141/141)
Adulterated samples	100 (47/47)	100 (141/141)

Table 4: Data of external validation from SIMCA models.

Samples	Specified material[a]	Identified material[b]	Total distance[c]	Residual distance[d]
1-23	Grapeseed oil	Grapeseed oil	0.42-0.74	0.75-1.12
1-23	Sunflower oil	Sunflower oil	0.13-0.81	0.39-1.24
1-23	Soybean oil	Soybean oil	0.56-0.83	0.78-1.15
1-23	Adulterated oil	Adulterated oil	0.28-0.85	0.35-1.02
1-23	Chia oil	Chia oil	0.16-0.71	0.22-1.01
1-23	Sunflower oil	Sunflower oil	0.13-0.98	0.17-1.26
1-23	Soybean oil	Soybean oil	0.18-0.62	0.23-0.65
1-23	Adulterated oil	Adulterated oil	0.75-0.96	0.86-1.63

[a]Specified material of validation; [b]Identified material by SIMCA model; [c]Total Distance (indicates if the sample was identified correctly (should be less than 1); [d]Residual Distance (should be less than 3)

The validation of the SIMCA model revealed that it can appropriately identify pure samples and adulterated samples (Table 4). Statistical values established limits (total distance ratio ≤ 1 and residual distance≤ 3), the above indicates that SIMCA models properly identified sunflower and soybean oils as adulterants with a 99% confidence limit.

Predictive models

The quantitative models were developed using the fingerprint region (1400-600 cm^{-1}) because this region presented a good correlation between absorbance FT-MIR and the percentage of adulteration in samples. Table 5 shows the statistics for calibration results in the developed predictive models.

PLS algorithm develop the best predictive models in comparison to the ones developed with PCR algorithm. For

Table 5: Calibration data to predict adulterants (sunflower oil and, soybean oil) in grapeseed oil and, chia oil.

Calibration set	Algorithm	Parameter (%)	Calibration (n = 47)			Validation (n = 23)	
			Factors[a]	R^2c[b]	SEC[c]	R^2v[d]	SEP[e]
GSS	PLS	Grapeseed oil	5	0.9695	2.25	0.9901	2.31
		Sunflower oil	7	0.9614	2.48	0.9915	2.79
		Soybean oil	7	0.9551	2.87	0.9906	3.13
	PCR	Grapeseed oil	20	0.9096	4.08	-	-
		Sunflower oil	23	0.8922	4.19	-	-
		Soybean oil	20	0.8255	4.34	-	-
CSS	PLS	Chia oil	8	0.9748	2.59	0.9925	2.31
		Sunflower oil	6	0.9595	2.22	0.9958	2.25
		Soybean oil	6	0.9549	2.33	0.9964	2.36
	PCR	Chia oil	6	0.8902	4.46	-	-
		Sunflower oil	18	0.9009	2.76	-	-
		Soybean oil	21	0.8863	4.79	-	-

GSS: grapeseed-sunflower-soybean oils; CSS: chia-sunflowers-soybean oils; [a]Factors; [b]R2c: coefficient of determination (calibration); [c]SEC: standard error of calibration; [d]R2v: coefficient of determination (validation); [e]SEP: standard error of prediction.

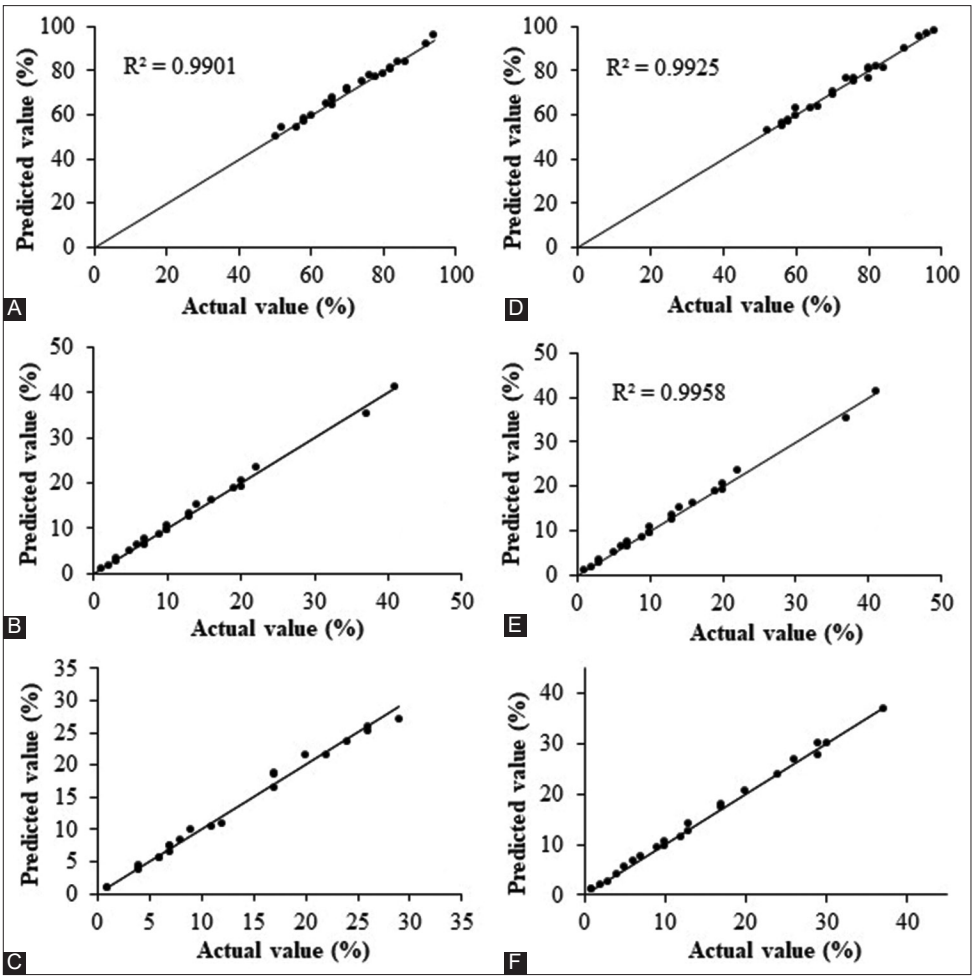

Fig 4. Plots of predicted values versus actual values for the validation samples determined by the PLS algorithm: a) grapeseed oil; b) sunflower oil; c) soybean oil; d) chia oil; e) sunflower oil; f) soybean oil.

the model that predicts adulterations in grapeseed oil, PLS algorithm presented a number of factors between 5 and 7. Factors must be ≤ 50% the number of the calibration data in order to avoid over-fitting (Beebe et al., 1998). In this sense, the number of factors obtained by the model that predicts adulterants in grapeseed oil agrees with the established. R^2c values were between 0.9551 and 0.9695. A value of $R^2c \geq 0.91$ indicates "excellent" quantitative information, meaning, the models presented excellent correlations between the real values and predicted values in each adulterant (Tamaki and Mazza, 2011). SEC data (2.25 – 2.87) indicate a small error in regression (PerkinElmer, 2014b). There is a similar tendency present in the model that predicts adulterations in chia oil because the PLS algorithm presented factors between 6 and 8, R^2c between 0.9549 and 0.9748 and SEC values between 2.22 and 2.59.

The calibration results for both developed models prove the capability of the PLS algorithm to predict and quantify the percentage of adulterants (sunflower, and soybean oils) in grapeseed and chia oil in the interval of 2-50% (w/w).

The above is reflected in the external validation of the models. Table 4 also shows R^2v and SEP values that were used for assessing the performance of the models developed. R^2v values of external samples were above of 0.99 for both models. SEP values were between 2.25 and 3.13. According to Beebe et al. (1998), models must have a high R^2v, and low SEC and, SEP values. Based on the above, the models that predict and quantify adulterants (sunflower and, soybean oils) in grapeseed and, chia oils agree with the established.

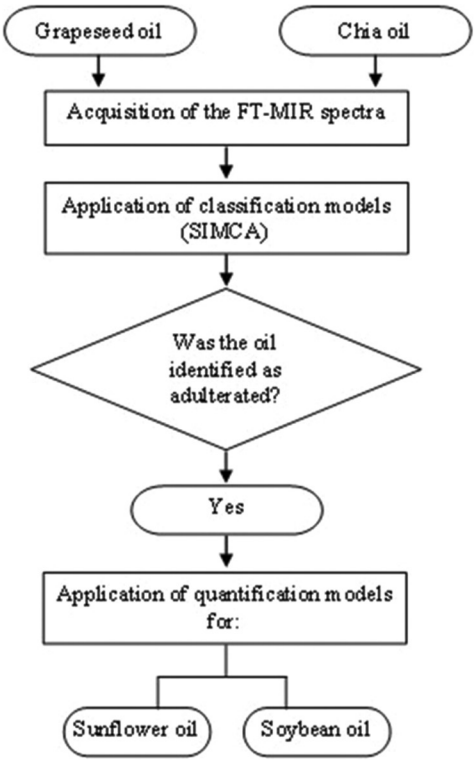

Fig 5. Flow diagram for the application of chemometric models.

Fig. 4 shows a good correlation ($R^2v \geq 0.99$) between real values and, predicted values of external samples used to validate all models. $R^2v \geq 0.99$ indicate the predictive ability of the models to predict all adulterants (sunflower, and soybean oils) in grapeseed oil and chia oil in the interval of 2-50% (w/w).

Finally, Fig. 5 shows the application of classification and quantification of the models developed. The models developed can identify and quantify adulterants (sunflower oil and soybean oil) in ternary mixtures in grapeseed oil and chia oil quickly, without using solvents or pretreatment of the samples.

CONCLUSIONS

The values of the physicochemical analysis of the oils (grapeseed, chia, sunflower, and soybean oils) are within the limits established by legislation regarding peroxide values and iodine values. Likewise, percentual values of Fatty Acids obtained by Gas Chromatography confirmed the authenticity of the oils used. The SIMCA models identified grapeseed oil, chia oil, and adulterants: sunflower oil and, soybean oil, with high confidence interval (99%). Likewise, prediction models quantified adulterants (sunflower and soybean oils) in grapeseed and chia oils in an acceptable interval (2-50%, w/w). The developed models could be applied in the industry to verify the authenticity of grapeseed and chia oils since these models are capable to predict the concentrations (2-50%, w/w) of the adulterants such as sunflower and soybean oils in ternary mixtures. It is recommended to develop more predictive models including other possible adulterants with the aim of building more robust models with the capability to predict other possible adulterants. In the future, other limits of detection and quantification of adulteration could be analyzed.

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Conflicts of interest

The authors declare no conflict of interest.

Authors' contributions

Conceptualization, C.B.V.-R., G.O.-R., and T.G.-V.; Methodology, C.B.V.-R., O.G.M.-M.; Formal analysis, C.B.V.-R., G.O.-R., and T.G.-V.; Investigation, C.B.V.-R.; Project administration, G.O.-R., and T.G.-V.; Resources, G.O.-R., and T.G.-V.; Supervision, G.O.-R., and T.G.-V.; Writing-original draft, O.G.M.-M.; Writing-review and editing, O.G.M.-M. All authors have read and agreed to the published version of the manuscript.

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