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Study of Oleaster Oil's Falsification by ATR-FTIR and Chemometrics Tools

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Abstract

This study aims to create a model of oleaster oil simply and reliably to detect adulteration, which presents a large danger that attacks the food sector and human health. For this reason, a study to detect the falsification of oleaster oil was carried out by Fourier-Transform Infrared Spectroscopy FTIR and chemometric method. The experimental samples are shared into two sets, 32 Training set, 8 Test set (4 calibration samples opposite one for validation), and a falsification interval of 1.5-40%. The treatment of infrared spectral results has been done by chemometrics techniques utilizing Partial Least Squares regression or Projection to Latent Structures (PLSR) and Principal Component Regression (PCR). The results show that the perfect falsification model of oleaster oil by olive-oil and soybean oil is illustrated in the spectral region 3050-2700 cm^{-1}, with R^2 of 0.999 from PLSR and PCR to soybean-oil, concerning olive-oil shows also the better results for the PLSR technical with R^2 of 0.995. The spectral and chemometrics results revealed an effective model that can detect adulteration whatever the type of adulterant used in this study (olive oil and soybean oil) with a percentage of adulteration ranging from 1.5% to 40%.

Keywords: Infrared spectroscopy; PLS; PCR; falsification; oleaster oil; olive oil; soybean oil.

1. Introduction

The olive is a tree known for its cultural and economic importance [1]; it is a cultivated form [2] with the olea europaea subsp. Europara var. Europaea [3] is the oldest agricultural crop in the Mediterranean basin [1]. Olive oil has a more important human use [4] because it is a source of aldehydes and hydrocarbons [5]. It is also characterized by its high resistance to oxidation [3]. On the other hand, virgin olive oil has excellent sensory properties and rich in unsaturated fatty acids, which has become healthy for consumers [6-9].

In addition to cultivated olive trees recognized worldwide, another olive variety is also very important and generates advantageous oil. It is the wild form of olive called Oleaster. Their nomenclature is Olea europaea subsp. europaea var sylvistris, characterized by a thorny bush with small fruits, a low oil content, a longer juvenile stage, has a strong capacity to withstand the harsh conditions of drought presence refers to a natural ecosystem [1-3].

Oleaster oil is a natural product that has been used in medicinal, cosmetic, and nutritional applications [10]. It has an interesting value because it contains significant amounts of the main fatty acids, monounsaturated (oleic acid), saturated (palmitic acid), polyunsaturated (linoleic acid), from a study done on 7 wild olive trees in Tunisia by Baccouri et al. in 2008 and which aims to determine the composition of this oil. The study also showed the most powerful antioxidant, which is tocopherol and phenols that improves the resistance to oxidation in oils [10]. Another study by Belarbi in 2011 found that oleaster oil harms cardiovascular risk and improves the plasma lipid profile of healthy volunteers [11].

Various other authors have discovered the

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interesting properties of oleaster; among them, it has a strong antioxidant activity because it contains significant amounts of antioxidants total tocopherol and total phenol, an antidote to certain poison and produces a good oil quality because it has major amounts of fatty acids and phenols [3, 12]. This importance results in a great demand that requires high quality and a high price [13]. For this reason and more profit, producers and sellers have directed towards the falsification of these oils, which attacks these beneficial properties and becomes a danger to consumer health [5, 13].

Adulteration can take different definitions depending on the literature. According to Dong sun, tampering changes a food product to another inferior, cheaper, and more available product [14]. According to Mariana, vegetable oils' falsification is repeated reuse of vegetable oil from the previous working and can also replace some components [8]. And for Souhassou, it is adding a non-automatic and cheaper substance to an original food product [15]. And then falsification, of food products in general and oils in particular as a major economic fraud that threatens consumers' health and can have serious consequences on public health [7, 8, 16]. To solve this problem, there are different methods and techniques to detect this falsification. Chemical indicators as the iodine value, the ranitidine value, and the Rancimat induction [14]. Analyses in the commercial laboratory [8]. Chromatography methods, like liquid chromatography. Vibration spectroscopy, mass spectroscopy, and nuclear magnetic resonance spectroscopy [5, 10]. All these methods distinguish forgery, but they need a long time, much money, complex preparations of the samples, and sometimes do not give good results [4, 6]. For this reason, researchers are directed towards the use of FTIR and chemometrics to detect and evaluate adulteration.

Each molecule is characterized by vibrational spectra, such as the infrared spectrum used to identify the molecules' functional groupings and obtained by the FTIR technique. This identification is carried out from the absorption of electromagnetic radiation during the chemical bonds' vibrations [16].

Fourier Transform Infrared Spectroscopy (FTIR) is a spectroscopic method that can determine and identify the size and number of particles and polymers [17]. He is considered an excellent analytical tool in the food field, mainly in olive oil dominates. It is a sensitive, fast, effective, non-destructive analytical technique and requires only simple sample preparation. It is used in the study of edible fats and oils, including the differentiation and classification of olive oils from different producing regions, giving the information about the chemical structure of olive oil samples, and evaluating the authenticity of olive oil, and the detection of their falsification by different falsifiers [4, 7, 18-23].

Chemometrics is a set of techniques based on the use of mathematical, statistical, and computational methods to extract useful information from chemical measurement data; it is used to interpret the results of experiments based on the construction and exploitation of a model of behavior using statistical tools [20]. Chemometrics is used to characterize and authenticate edible fats and oils [21], distinguish types of different geographical origins, genetic varieties of vegetable oils, and detect adulteration [4, 8].

In this context, the present study aims to detect oleaster oil's adulteration with healthy and therapeutic benefits. Due to its low abundance, people and producers have directed to mix it with other oils and mix oleaster fruits with cultivated olive fruits. The molecular composition of the oleaster oil by FTIR-ATR was first determined, and then chemometrics including Partial Least Squares regression or Projection to Latent Structures (PLSR) and Principal Component Regression (PCR) [24] were used to build an adulteration model that can detect the presence of adulteration and determine their concentration.

2. Materials and methods

2.1. Study zone

The study site and sampling stations' location is Tamchat Beni Mellal, Morocco (Fig. 1).

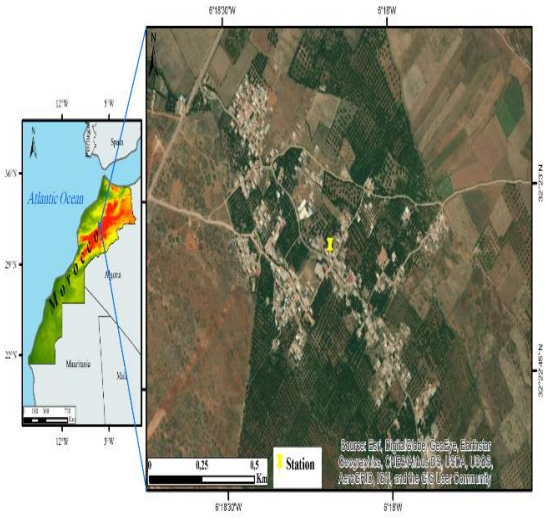

Fig. 1. Tamchat, Beni Mellal Morocco (Esri, DigitalGlobe).

2.2. Plant material

The olive fruits are black and size between 1-2 cm; they are harvested from the Tamchat region between 2 and 3 February 2019. The extraction method was as follows: the oleaster fruits were crushed and exposed to the sun for two days; after pressing the paste well to obtain a wild olive oil, for every 10.5 kg, 0.25 liters was obtained.

2.3. Sample preparations

Oleaster oil was weighed in grams; then, this sample was falsified by increasing soybean oil percentages. The same process does the falsification by olive oil. The mass percentage was calculated for each of the samples.

2.4. Analytical method

FTIR is a better analytical technique that requires only a simple sample preparation and gives reliable and relevant results. It is used to detect the falsification of oleaster oil by other oils. After reading the samples with FTIR, the samples are analyzed using chemometrics to detect falsification and create the model. A series of 32 oleaster oils were sampled for each case from the Beni mellal Moroccan region (Tamachat) during the harvest season 2019. All the samples belonged to the variety known as Marocain. The collection method, the storage conditions, and preservation were the same for all the samples. Samples were tested immediately after their production by FTIR in an analytical range of 4000 to 600 cm^{-1}. A group of 32 samples was randomly selected to constitute the calibration model, whereas the other 8 samples were chosen as a set for external validation and with a falsification interval of 1.5-40%.

The Spectrometer obtained the spectra of the samples (PerkinElmer) instrumented with Attenuated Total Reflectance accessory (ATR single reflection, Diamond, angle of incidence 45 incident C), DTGS detector, Globar source (MIR), and KBr Germanium separator, with a resolution of 4 cm -1 to 98 scans. The spectra were scanned in an absorbance range from 4000 to 450 cm^{-1}, and the spectra were processed with software. The analyzes were performed at room temperature. The recorded spectra of each sample of pure and adulterated oleaster oil are entered with the mass percentages calculated in Unscrambler Software.

2.5. Chemometrics techniques

PLSR means regression in the sense of partial least squares, also links a set of dependent variables Y, to a collection of independent variables X, when the number of variables (independent and dependent) is high. PLSR is the most frequently used regression method in chemometrics.

PCR is the method of linking variations in a response variable (variable Y) to variations in several predictors (variables X) for explanatory or predictive purposes. It is a two-step procedure that first breaks down an X matrix by PCA, then adjusts to an MLR model, using PC scores instead of the initial X variables as predictors.

3. Results and discussion

The infrared spectroscopic analysis from oleaster oil samples (OlO) and two falsifying olive oil (OO) and soybean oils (OS) are used in this work due to their low prices and lipid similarities [25]. Results from the spectral curves observed in Fig. 2. This figure illustrates the typically FTIR spectra of these oils (OlO, OO, and OS) in the spectral region comprised between 4000 cm1 and 450 cm^{-1}, which appeared almost similar among the other spectra of the different analytical works carried out on olive oil [7, 13, 22-27].

A comparison between the oleaster oil spectrum with the two falsifiers shows a complete overlap between the OlO and OO spectrum (B), whereas there are only two differences compared to the OS at the spectral region level 2896-2796 cm^{-1} and 1200-800 cm^{-1} (A). This difference in the two spectra from oleaster oil and soybean oil was caused by the difference in the two oils' constituent fatty acids' chemical structure.

Visual observation shows a tendency towards these spectra's general appearance as they appear to be from different spectral regions with different spectral peaks. For the spectral region 4000-3100 cm^{-1} and 2800-1800 cm^{-1}. That is to say, the oils studied do not have infrared absorption in these regions. For spectral region 3000-2800 cm^{-1}, there are three particulate peaks in this region, two peaks having 2900 and 2840 cm^{-1} who characterized by the existence of the molecular functions C-H aliphatic group and C-H aromatic group, and another at 3006 cm^{-1} which informs the compound of the stretching C-H [16, 27]. Concerning the spectral region of 1800-1700 cm^{-1}, there is a vibration band around 1746 cm^{-1}, resulting in the group (CO) triglyceride or a functional group carbonyl ester of the triglycerides [16, 26, 28]. The spectral region of 1500-500 cm^{-1} is characterized by different molecular functions, including

alkane/alkyl/alkene/alkyne C-H functions, aromatic C=C-C, alcoholic groups O-H and C-H [16], and also different C-O-C and CH bonds [7, 23]. In general, in Fig. 2, there are about nine small spikes, which indicate the multitudes of molecular groups extracted from the corresponding wavenumbers. The largest of 1160 cm^{-1} (-C-O stretch and $–CH_2$ flexion) and the smallest of 600 cm^{-1}. However, some shoulders were raised between 850 to 1100 cm^{-1}.

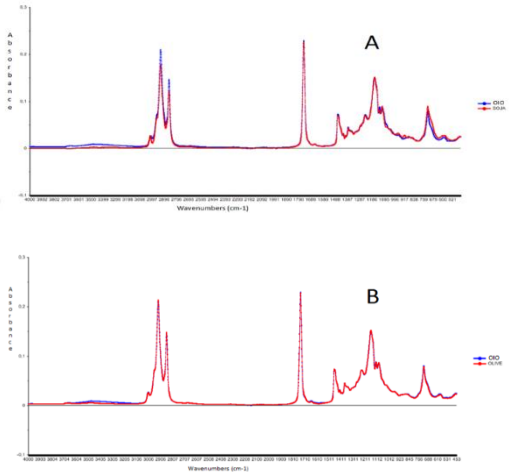

Fig. 2. Spectra from oleaster oil about oil soybean (A), oil olive (B).

Table 1
The functional group of oleaster oil obtained from the FTIR [29].

Range (cm^{-1})	Functional group	Mode of vibration
3006	C–H (trans)	Stretching
2946–2881	–C–H (CH_2)	Stretching (asym)
2881–2782	–C–H (CH_2)	Stretching (sym)
1795–1677	–CO (ester)	Stretching
1486–1446	–C–H (CH_2)	Bending (scissoring)
1382–1371	–C–H (CH_3)	Bending (sym)
1290–1211	–C–O	Stretching
1211–1147	–C–O	Stretching
1128–1106	–C–O	Stretching
1106–1072	–C–O	Stretching
754–701	$–(CH_2)_n–$	Rocking

The interpretation of the spectral bands obtained from Dr. Lerma-García's bibliographic data provides information on the molecular structure and functional groups of the studied oil samples, illustrated in Table 1. The spectra are predominant by two main peaks of 2900 cm^{-1}, which are very fine and mean the functional group -CH (CH_2) and the peak of 1720 cm^{-1}, which is very long, very delicate, and illustrates the functional group -CO (ester). According to the literature and various studies carried out on the authentication of olive oil. In particular, the realization of an agreed and more practical model to determine the presence of falsification and to specify the falsifier's concentration. Among these studies, some have based on the use of FTIR only from a calibration curve established from a thorough analysis of spectral results [13]. Other studies are based on FTIR, coupled with chemometrics, to create the right model. A discriminating study carried out on olive oil adulterated by different vegetable oils (rapeseed oil, sunflower oil, cottonseed oil) by the PLSR algorithm and with the OSC software has 10% to determine the effectiveness of the model regardless of the type of adulterant [23]. Another chemometrics study was based on the use of PLSR and PLS-DA to achieve a good model of adulteration of virgin olive oil falsified by soybean oil and sunflower oil with a concentration range between 1-24% by weight [7]. There is also, in this context, the analysis made by Hirri on virgin olive oil falsified by another old olive oil by using the chemometric methods PLSR and PCR to establish a model able to know the concentration of the falsification [27].

FTIR spectral analysis provides infrared spectra that detail the chemical composition of oils and the different functions' molecular rotations. This analysis did not make it possible to determine the adulterant and the concentration of falsifier in oils accurately and reliably because all oils have the same spectral curves. Therefore, these spectra, coupled to chemometrics, can create a good model of falsification. In this work, the FTIR spectra were thermometrically coupled to the least square algorithms (PLSR), main component regression (PCR), and Unsclamber software because the FTIR spectra of all oils after their analysis have identical peaks that indicate the same functional groups.

The chemometrics analysis, by PLSR and PCR, was obtained from a linear method that links the absorbance (by FTIR) with the corresponding (experimental) mass concentrations. The results from the calibration factors (RMSE with two values: the calibration error RMSEC in blue and the expected prediction error in red, and R square: R-value of calibration square in blue and validation value in red), and validation factors (R^2 person which indicates a correlation on a positive scale between 0 and 1 and RMSEP expressing the dispersion of the validation samples around the regression line). A good model is one that has the highest R^2 value and the lowest RMSEP value.

The FTIR spectra thermometrically processed by PLSR, PCR, and Unsclamber software of 32

calibration samples and 8 validation samples of oleaster oil falsified by soybean oil and olive oil gave the results shown in Figs. 3, 4, 5, and 6. Also, chemometrics, including PLSR and PCR, measure and distinguish the difference between spectra by the regression curve, where very elongated spikes well illustrated the difference between the studied oil and their falsifier with wavelengths between 3050-2700 cm^{-1} for soybean oil (Figs. 3A and 4A), and olive oil (Figs. 5A and 6A).

PLSR and PCR thermometrically treat the spectral difference established by the regression model, and results were shown in Fig. 3B and 4B for soybean oil, 5B and 6B for olive oil, which creates a calibration model capable of determining falsification. These figures give calibration parameters (R-square, RMSE) that have been grouped in the following Tables 2 and 3. RMSE is another chemometric parameter that displays two values. The first represents the RMSEC calibration error, and the second displays the expected prediction error based on the validation method used. A small RMSE gives a better model.

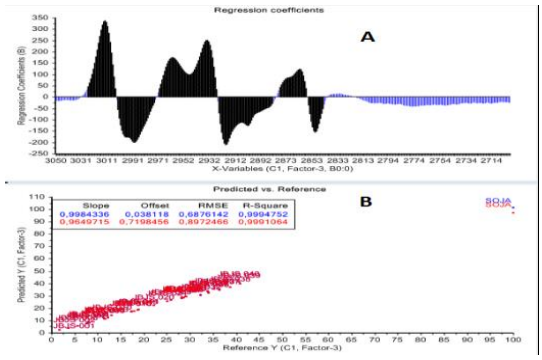

Fig. 3. PLSR for oleaster oil falsified by soybean oil in 3050-2700 cm^{-1} of the calibration set.

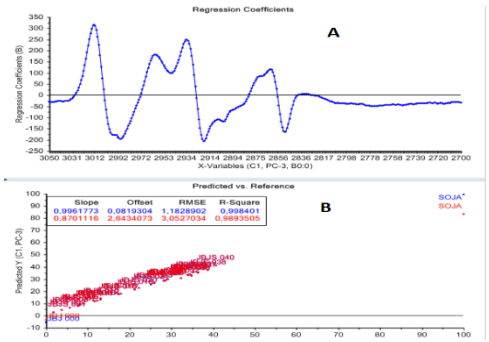

Fig. 4. PCR for oleaster oil falsified by soybean oil in 3050-2700 cm^{-1} of the calibration set.

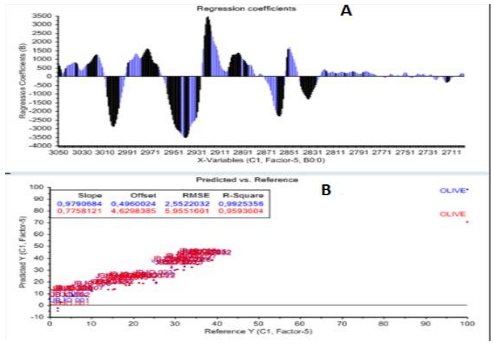

Fig. 5. PLSR for oleaster oil falsified by olive oil in 3050-2700 cm^{-1} of the calibration set.

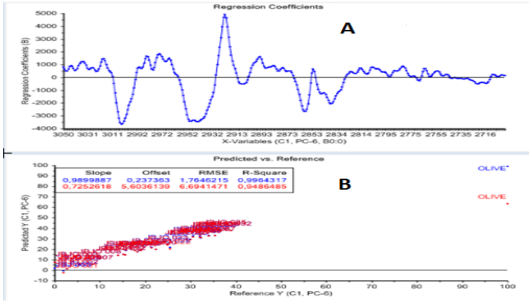

Fig. 6. PCR for oleaster oil falsified by olive oil in 3050-2700 cm^{-1} of the calibration set.

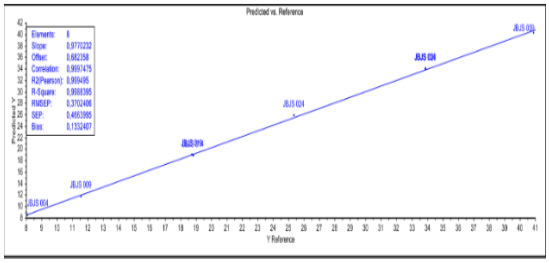

Fig. 7. Model of oleaster-soybean in 3050-2700 cm^{-1} (PLSR) of the test set.

Table 2 :Parameterized calibration and validation of oleaster-soybean (PLSR and PCR).

Wavenumber (cm^{-1})		4000-450	3200-450	1900-450	1500-450	3050-2700
		0.9976	0.9971	0.9954	0.9946	0.9994
	R-square	0.9977	0.9965	0.9937	0.9931	0.9993
PLSR		1.1909	1.3200	1.6674	1.7980	0.5744
	RMSE	1.2749	1.4810	1.9436	2.0361	0.6433
	R^2 Pearson	0.9634	0.9577	0.8929	0.8814	0.9994
	RMSEP	2.4420	3.0645	5.3668	5.7104	0.3702
		0.9967	0.9965	0.9918	0.9926	0.9994
PCR	R-square	0.9963	0.9958	0.9905	0.9914	0.9992
	RMSE	1.4540	1.4482	2.2275	2.1094	0.5866
		1.4905	1.5837	2.3904	2.2751	0.6544
	R^2 Pearson	0.9350	0.9479	0.8444	0.8518	0.9994
	RMSEP	3.2905	3.4284	6.6890	6.5650	0.3776

Table 3 :Parameterized calibration and validation of oleaster-olive (PLSR and PCR).

Wavenumber (cm^{-1})		4000-450	3200-450	1900-450	1500-450	3050-2700
		0.9818	0.9863	0.9937	0.9953	0.9925
	R-square	0.9786	0.9838	0.9892	0.9900	0.9593
PLSR		3.3161	2.8765	1.9448	1.6702	2.5522
	RMSE	3.5959	3.1286	2.5498	2.4522	5.9551
	R^2 Pearson	0.9509	0.9654	0.9870	0.982	0.9958
	RMSEP	2.6446	2.2406	1.5077	1.8357	1.7895
		0.9911	0.9857	0.9923	0.9928	0.9964
PCR	R-square	0.9884	0.9833	0.9880	0.9883	0.9486
	RMSE	2.3078	2.9393	2.1569	2.0805	1.7646
		2.6380	3.1700	2.6868	2.6581	6.6941
	R^2 Pearson	0.9840	0.9638	0.9847	0.9767	0.9897
	RMSEP	1.5758	2.3002	1.6238	2.0258	1.6076

Depending to the division of the spectral range 4000-450 cm^{-1} according to the spectral difference obtained by the spades, there is the obtaining of an R-square close to 0.999 in the regions of 4000-450 cm^{-1}, 3200-450 cm^{-1}, 1900-450 cm^{-1}, 1500-450 cm^{-1} and the RMSE for calibration and validation close to 2 for the oleaster-soybean (Table 2). To obtain a good R^2 and a minimum value of RMSE and RMSEP, the spectral region of 3050-2700 cm^{-1} was chosen to realize the oleaster-soybean's chemometrics model.

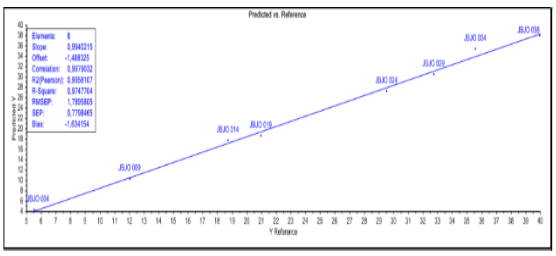

Fig. 8. Model of oleaster-olive in 3050-2700 cm^{-1} (PLSR) of the test set.

The spectral region of 3050-2700 cm^{-1} after their treatment with PLSR and PCR gives a better R^2 of 0.999, a small RMSE (0.5744-0.6433), and an RMSEP min of 0.3702 (Table 2).

So it is the right model can use for detection of forgery of oleaster-soybean (Fig. 7). Concerning oleaster-olive adulteration, the same division was made to look for a good correlation (Table 3). Table 3 gives results near to 0.999 for R square and values between 2 and 5 for RMSE about oleaster-olive. After the comparison of R^2 and RMSEP (Table 3) spectral regions, the correct model is chosen that reaches the conditions of good chemometrics modeling is that of the spectral region 3050-2700 cm^{-1} for PLSR who has a high R^2 value (0.995), and RMSE (2.5522-5.9551), and a low RMSEP value (1.7895). The oleaster-olive falsification model was illustrated in Fig. 8.

The R^2 (0.999 and 0.997) has been found across various studies that have been done to detect adulteration of olive oil. Rohman et al.'s work result in an R^2 of 0.998 (PLS and PCR) through the falsification

of extra virgin oil by palm oil in a spectral range of 1500-1000 cm^{-1} an adulteration percentage of 1-50% [20]. In another study by Oussama et al., there is an R^2 = 0.997 (PLS) of falsification of olive oil by sunflower and soybean oil (3035-670 cm^{-1}) with a percentage of falsification 1-24% [7]. Hirri et al. also found an R^2 of 0.999 (PLSRet PCR) in a study on recent olive oil and their falsification by an old olive oil within a spectral range of 3100-600 cm^{-1} [21]. Another study with an R^2 of 0.9870 focuses on determining olive oil's geographical origin by Lin et al. using visible and near-infrared spectroscopy with Direct Orthogonal Signal Correction-Genetic Algorithms-PLS [30]. Sun et al. employed FTIR with PLS to quantify extra virgin olive oil with the other edible oils used as falsifiers; they obtained an R^2 between 0.98-0.99 and RMSE between 1.9-9.5% depending on the nature of the falsifier [28-31]. Another recent study of the quantification of EVOO with oils from different seeds showed that the use of chemometrics has pertinent advantages in the detection of adulteration through the application of chemometric quantification with PLS, which gave results of R^2 = 0.99 and a SEP = 0.623, with a percentage of adulteration ranging from 5 to 95% [32]. As well as, the rapid quantification results of EVOO adulterated (5-45%) by vegetable oils (soybean, peanut, sunflower, canola, corn) showed better values obtained by PLS with R^2 = 0.994 in the range of 1500-800 cm^{-1} [33].

4. Conclusions

In this study, manipulation of ATR-FTIR coupled with chemometrics has successfully solved the falsification of oleaster oils with efficiency by producing a very promising model to detect and identify the concentration of these oils, whatever of the adulterant, and the aspect of adulteration. The results obtained from this study showed that FTIR and chemometrics could detect and evaluate the authenticity of oleaster oil tested by two adulterants easily and reliably. The spectral analysis gives a molecular and functional explanation of this oil and shows a spectral similarity with olive oil in the spectral range of 4000-450 cm^{-1} and with soybean oil except for two spikes in 2896-2796 cm^{-1} and 759-521 cm^{-1}. The percentage of adulteration the olive and soybean oils in oleaster oils ranging from 1.5% to 40% (v/v). It is going up to 40% because in adulteration with olive oil, cheaters have a wide range of possible adulteration. That is due to an approach similarity between the two oils in smell, taste, color, and even chemical composition in which the FTIR spectra of these oils are approaching. Simultaneously, the chemometrics analysis, which PLSR and PCR have done, gives modeled results that differ from the R^2 and RMSEP in the spectral intervals according to the differentiation of the obtained peaks.

Moreover, according to this finding, the falsification model in the interval 3050-2700 cm^{-1} considering their R^2, which is 0.999 for oleaster-soybean and 0.995 for oleaster-olive, has been chosen. The use of FTIR, combined with chemometrics, has shown its effectiveness and usefulness in the adulteration of oleaster oil and other food products [33]. After this chemometric study of the adulteration of oleaster oil, the adulteration with other falsifiers and their classification will be studied, as well as the determination of the variability and the classification of oleaster oil samples depending on the agricultural regions, as well as the determination of their Physicochemical parameters in an analytical and chemometric way because of their sanitary and therapeutic importance.

Conflicts of interest

There are no conflicts to declare.

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