



Authenticating Edible Oils Using Fourier Transform Infrared Spectroscopy: A Review

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ABSTRACT

Oil authentication has been widely discussed in recent years. One of the issues is the usage of gutter oil. This happened in China where many of the street foods were prepared using oils from sewage, gutters, and restaurant fryers. Other concerning issues including the adulteration of high-quality edible oils with cheaper oils and fresh palm oil with recycled cooking oil are common problems related to oil fraud. This may provoke the safety and the rights of public consumers. Hence, advanced, efficient, and rapid technology such as Fourier Transform Infrared Spectroscopy (FTIR) is needed to overcome the limitations of other technologies such as differential scanning calorimetry (DSC), gas chromatography-mass spectrometry (GC-MS) and high-performance liquid chromatography (HPLC) in analysing edible oils' quality parameters, authentication, safety, stability and in foods related to oils. This review discusses the uses of FTIR in the analysis of edible oils and their authentication.

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1. INTRODUCTION

Infrared (IR) spectroscopy is an archaic technique for obtaining information on complex structures, such as identifying unknown samples, determining sample consistency, and determining the number of components in a mixture [1]. Sir William Herschel discovered the IR radiation in 1800 [2]. Later, in the 20th century, William W. Coblentz's efforts led to the establishment of IR spectroscopy as a key analytical method in academic and industrial labs. The introduction of a Fourier Transform Infrared (FTIR) spectrometer using an interferometer by Albert Michelson in 1891 can convert a spectrum to a mathematical operation with the help of Lord Rayleigh. This mathematical operation was discovered by Fourier, a French mathematician in the 1820s.

FTIR is evolving and it is now recognized as a technique for examining the magnitude of infrared radiation at a specific wavenumber that is absorbed or transmitted by a sample, yielding the sample's molecular fingerprint [3][4]. A

molecular fingerprint ensures that there are no molecular structures that can produce the same infrared spectrum. Hence, FTIR has commonly been used in several types of analysis such as fats and oils analyses, forensics, and environmental testing. The development of FTIR is due to the limitations of IR in sample analysis. The dispersive type of IR only measures the individual frequencies that pass through the sample rather than all infrared frequencies [5]. FTIR overcomes this constraint by using an interferometer to generate a signal that incorporates all of the infrared frequencies encrypted within it. FTIR offers a rapid and reliable method without the need for sample preparation and a large number of solvents or reagents [6]-[8]. In comparison to other methods such as differential scanning calorimetry (DSC) [9], gas chromatography-mass spectrometry (GC-MS) [10][11] and high-performance liquid chromatography (HPLC) [12][13] that are time-consuming, require skilled

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operators, expensive, destruction of sample and too laborious [3][6][14].

FTIR is commonly used to verify the consistency and validity of edible oils and the items that contain them. One of the major concerns was when China started using gutter oil in 2000 at a low price [15]. According to Rohman [16], the detection of adulterants is not the only authentication analysis in edible oils but includes other analyses such as plant species geographical identification, the production of the edible essential oils and the detection of foreign materials in the oils. Hence, the analysis will help food manufacturers to ensure that the edible oils sold are not adulterated and their quality meets the standards and specifications established by national authorities. In addition, adulteration of edible oils may cause serious health problems among unwary consumers [17]. FTIR has detected the adulteration of edible oils by other forms of vegetable oils in previous studies. FTIR with chemometrics analysis has managed to identify the adulteration of sesame oil with hazelnut, canola, and sunflower oil [18], extra virgin olive oil with peanut oil and rapeseed oil [8][19], avocado oil with soybean and corn oil [6], and fresh palm oil with recycled cooking oil [15][20]. Thus, the objective of this review paper is to present an overview of the uses of FTIR in edible oil analysis and authentication as an alternative method to DSC, GC-MS, HPLC and IR spectroscopy.

2. EDIBLE OIL ANALYSES IN FOOD

Food adulteration has widely been practiced among unethical manufacturers and traders. One of the most consumed foods is canned products. Because of the health benefits of polyunsaturated fatty acids (PUFAs), demand for fish has increased [21]. Fish canned products such as canned tuna have been opted to preserve tuna from spoilage, provide longer shelf life while maintaining its nutrition and qualities and can be readily eaten without much hassle [22]. Thus, edible oil is chosen as the liquid medium of canned tuna. Dominguez-Vidal [21] stated that edible oil such as olive oil helps in preserving tuna as well as make the product more palatable. Several studies are listed in Table 1.

Past studies showed Dominguez-Vidal [21] used FTIR to authenticate olive oil that was added to the canned fish. They discovered that regions 3270 cm^{-1} (N-H stretching, amide A), 1621 cm^{-1} (C=O stretching, amide I) and 1539 cm^{-1} (N-H bending coupled with C-N stretching, amide II) dominated the spectrum in the span of 4000-450 cm^{-1} . All the absorption bands above indicated the characteristics of proteins and were also found to overlap with water content released from fish flesh to packing oils at 3370 cm^{-1} , 1640 cm^{-1} and 692 cm^{-1} . Not only that but lipids can also be seen present in the fish at 2960 cm^{-1} , 2923 cm^{-1} , 2854 cm^{-1} , 1743 cm^{-1} , 1456 cm^{-1} and 1160 cm^{-1} . Then, they compared with a typical spectrum of packing oil such as olive oil and rapeseed oil where the differences can be observed at 3006 cm^{-1} (C-H stretching in cis olefinic double bonds), 710 cm^{-1} (CH in cis double bonds) and between more complicated bands at a region of 1000 and 800 cm^{-1} . By further analysis using the principal component analysis (PCA), there was a clear separation of different grades of olive oils with other seed oils with a variance of 98.1% except for high-oleic-sunflower oil. This was because high-oleic-sunflower has a similar composition of monounsaturated oleic acid to olive oil.

Another popular type of fish is Patin fish (*Pangasius micronemus*). There was a high demand for this freshwater fish, especially in Indonesia since they are rich in omega-3 fatty acid sources and good for human health [23][24]. Omega-3 helps prevent heart disease, Alzheimer's, and cancers [27]. Rosiana Putri [24] studied the adulteration of Patin fish oil (0-100% (v/v)) with palm oil using FTIR since both oils have analogous structures and it is easy to adulterate higher price oil with cheaper price oil to gain profits. However, the mixing will decrease the quality of Patin fish oil. Hence, methods to authenticate Patin fish oil are needed to protect consumers from fraudulent practices. Rosiana Putri [24] found differences in the spectrum between Patin fish oil and palm oil at 2954 cm^{-1} and fingerprint area (1500-650 cm^{-1}). Palm oil was found to have no peak at 2954 cm^{-1} (CH₃ asymmetrical stretch) and 721 cm^{-1} (rocking vibration of methylene -CH₂). As a result, these two regions can be used to detect Patin fish oil adulteration with palm oil.

Milk is a good source of protein, calcium, potassium, and phosphorus, and it is particularly essential for babies, teenagers, pregnant women, and the elderly [28][29]. Therefore, consumption of adulterated milk and cheese may lead to health risks among consumers. In the recent past, Dankowska [30] presented a study to monitor the original cheese adulterated with plant oils using fluorescence spectroscopy. However, since nearly all of the methods are time-exhausting, require handling skills and expertise, and involve sample preparation and hazardous chemicals, the combination of advanced IR spectroscopy and chemometrics analysis has emerged as a promising approach [31][32].

Leite [33] reported on a study about differentiating pure soybean oil and pure butter oil and adulterated soybean oil in butter cheese using FTIR (0-100% (w/v)). The spectra were measured from the 4000-400 cm^{-1} range for pure butter oil, pure soybean oil and butter cheeses. Similar spectra were seen with minor differences related to the intensity of the bands. Major peaks can be seen from 3600-2750 cm^{-1} and 1800-625 cm^{-1} . Since the spectra of cheese were complicated due to the presence of variations in functional groups, it was difficult to discover adulterated cheeses. However, when butter oil and soybean oil in cheese were compared to cheese without fat, a clear differentiation can be seen from 3600-3050 cm^{-1} and 1000-400 cm^{-1} . Furthermore, due to the presence of various forms of fat, such as soybean oil and butter oil, the absorbance intensities of adulterated butter cheese were higher than non-adulterated butter cheese, except for adulteration concentrations of 80%, 90%, and 100%. The intensities at these concentrations decrease in absorbance due to the higher level of soybean oil in the cheese at region 2922 cm^{-1} , 2852 cm^{-1} , 1743 cm^{-1} , 1464 cm^{-1} , 1160 cm^{-1} and 721 cm^{-1} . As a result, the higher the amount of soybean oil in the cheese, the more oil is released from the cheese protein network, resulting in a thin film of soybean oil on the surface. The thin film formation interrupts the MIR measurement. Furthermore, the band exhibited maximal absorbance at 3007 cm^{-1} (stretches of -C=C-H in unsaturated fatty acids) at concentrations of 0% and 10%, while from 20% to 100%, the band moved to 3009 cm^{-1} . Hence, it was concluded that, as the band shifted and increased, the unsaturated fatty acids in soybean oil were present. 3600-3050 cm^{-1} represents the hydroxyl group hence it was strong at 0% concentration. However, as the concentration increases, the band gradually disappears due to the migration of soybean oil on the surfaces.

Authors also related moisture content with the band intensity of amide I and amide II where when the oil content increased in the cheeses, the intensities of those decreased instead. To put it more simply, as the fat content drops, moisture and protein content rise.

The findings abovementioned underscore the significance of FTIR techniques in combating food adulteration, protecting consumer health, and maintaining the integrity of food products in the market.

Table 1. Applications of Fourier Transform Infrared Spectroscopy (FTIR) in Edible Oil Authentication in Food Products

Product	Issues	Sub-Issues	Aim of study	References
Butter cheese	Adulteration	Addition of soybean oil	To differentiate soybean oil in butter cheese adulteration	[33]
Canned fish packing oil	Adulteration	Addition of other vegetable oils	Detection of seed oils from olive oil in a canned fish packing	[21]
Patin fish oil	Adulteration	Addition of palm oil	Authentication of patin (<i>pangasius micronemus</i>) fish oil adulterated with palm oil	[24]
Milk Fat	Adulteration	Addition of palm oil	Authentication of palm oil in milk fat	[25]
Butter	Adulteration	Addition of margarine	To distinguish pure butter samples from adulterated butter samples and to assess the degree of margarine adulteration in butter samples.	[26]

3. EDIBLE OIL AUTHENTICATION COMBINED WITH CHEMOMETRICS ANALYSIS

One of the most adulterated food ingredients is edible oil. Oils and fats are essential components of the human diet because they provide nutrition and serve as a precursor to prostaglandin hormones [34]. Due to the increase in the demand for edible oils consumption, these sources are always under continuous threat from adulteration [35]. Hence, precautionary steps need to be taken to counter this problem, which is the authentication of edible oils. Authentication can be classified based on 1) geographical origin, 2) addition of foreign substances 3) adulteration with different types of edible oils, and 4) adulteration with used cooking oil. The most common authentication issues are authentication of olive oils due to their benefits, demands and high price in global markets [8][36][37]. This review paper presented an overview of different types of edible oils with different authentication issues detected using FTIR and chemometrics (Figure 1).

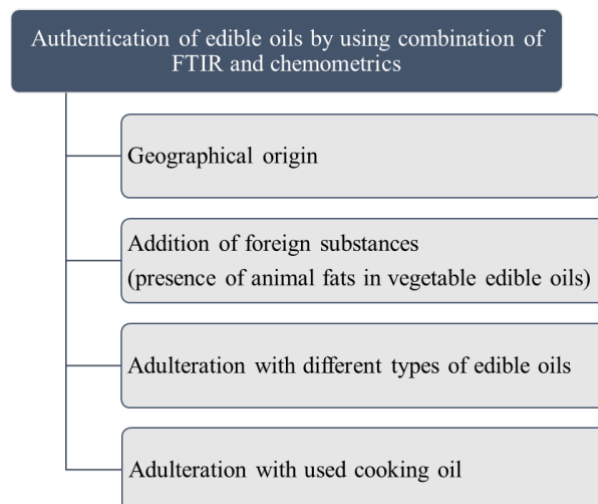


Fig. 1. Different authentication issues were detected using combination of FTIR and chemometrics

3.1 Geographical origin

The Olive Oil Source (1998-2019) mentioned that olive was native to Asia Minor and later was spread to the Mediterranean 6000 years ago. People around the world consume olive oil thanks to its distinct flavour [4]. While monovarietal olive oil is said to be expensive, it is in high demand [36]. Therefore, they are susceptible to suffer adulteration and mislabelling practices. Olive oil analysis has been using methods such as GC-MS [38], HPLC [13], capillary electrophoresis (CE) [39], and mass spectrometry (MS) [40].

Hirri [41] used FTIR and PLS-DA to predict the commercial-grade content of different Moroccan olive oils (VOO) based on their geographical origin. Spectra of 70 samples were scanned from 4000-600 cm⁻¹ and were measured in absorbance mode. The regions found were 3006 cm⁻¹, 2920 cm⁻¹ and 2852 cm⁻¹, 1743 cm⁻¹, 1463 cm⁻¹, 1377 cm⁻¹, 1237 cm⁻¹, 1161 cm⁻¹, 1118 cm⁻¹, 1096 cm⁻¹ and 722 cm⁻¹. Later, PLS-DA is used based on the spectral range 3100-600 cm⁻¹ as X-variables while four types of VOO grades as Y-variables. This was to classify samples into four grades of VOO: extra virgin olive oil (EV), virgin olive oil (V), ordinary virgin olive oil (OV) and lampante virgin olive oil (LV). The results were found to be only satisfactory for LV with negative scores while EV, V and OV grades were scattered together on the right side of positive scores. From the analysis, the VOO grades can be distinguished based on their polyphenol contents. EV, V, and OV contain a high level of polyphenols, less acidity and more peroxide index compared to LV. Although each grade has different polyphenols, acidity levels and peroxide index, the best quality among EV, V, and OV cannot be determined. As a result, FTIR combined with PLS-DA was able to effectively differentiate VOO based on its grades.

Messai [42] tried to differentiate olive oil (OO) samples from non-OO based on the FTIR spectra range from 3588-650 cm⁻¹. 70 OO samples originated from European and American countries and 41 edible oils were used for this study. Hierarchical cluster analysis (HCA) was then used to demonstrate three clusters that separated OO samples from

two other oil groups. HCA is one of the chemometrics analyses, which is used to classify the heterogeneous population into well-separated homogeneous groups called clusters [43]. Even though all 70 OO samples were placed together in the third cluster, one high oleic sunflower, one safflower, and two peanut oils were included in the group which is non-OO samples. The first and second clusters, on the other hand, included flaxseed oils and mostly non-OO samples, respectively. The authors suggested that some non-OO have similar compositions and profiles with OO samples. This study has proven that HCA can use all the information obtained from the FTIR spectra to distinguish OO from other types of edible oils. The main spectral that contributed to the differentiation was spectral bands of triglycerides containing unsaturated fatty acids.

Messai [42] also presented the application of FTIR with other chemometrics analysis, which was support vector machines (SVM). SVM is a supervised method for classifying unknown features into one of two categories. In this study, SVM is used to differentiate between Italian and not Italian OO and Ligurian and other Italian regions. FTIR spectra range 4000-600 cm^{-1} was used to characterize the spectra for 668 OO samples. However, region 2400 cm^{-1} and 2250 cm^{-1} was removed due to the presence of atmospheric carbon dioxide in this region. The ends of the range were also removed since they contain mainly noise. Thus, only regions 3000-2400 cm^{-1} and 2250-700 cm^{-1} were used. The combination of FTIR and SVM analysis allowed the samples to be differentiated into two groups.

Uncu and Ozen [44] reported on an investigation the geographical differentiation of monovarietal olive oil (total 54 samples) taken from nine distinct locations (Eglenhoca (EH), Karareis (RS), Karapinar (KP), Barbaros (BR), Gulbahce (GB), Torasan (TR), Kuscular (KS), Ozbek (OZ) and Urla (UR)) of the west part of Turkey using FTIR coupled with PCA and PLS-DA analysis. The olive oil sample was obtained from the Erkençe variety grown in a small, local area of the Karaburun Peninsula of Turkey and its close vicinity. It has not been studied due to its high oil content and early ripening time. Thus, identifying the characteristics of this variety allows the correct geographical indication labelling. The spectra of 4000-650 cm^{-1} were obtained and were able to separate the samples depending on their growing location. The first PLS-DA model (model I) successfully differentiated EH and RS oils while KP and OZ oils showed unclear separation. In the second model of PLS-DA, there was better separation for other oil samples belonging to EH, RS, KP and OZ regions based on their infrared profiles. Model I have a PCA degree of fit, $(R^2) = 0.94$ and predictive ability $(Q^2) = 0.26$ while Model II has PCA, $R^2=0.97$ and $Q^2=0.43$.

In summary, the geographical origin of olive oil has been extensively studied using advanced analytical techniques such as FTIR spectroscopy coupled with chemometrics analyses. Various studies have successfully employed FTIR to differentiate olive oils based on their geographical regions, grades, and even identify potential adulteration. The application of chemometrics tools like PLS-DA and PCA has enhanced the accuracy of classification, allowing for the effective distinction of olive oil samples (Table 2).

Table 2. Application of Fourier Transform Infrared Spectroscopy (FTIR) in Olive Oil Authentication and Traceability

Product	Issues	Sub-Issues	Chemometrics	Aim of study	References
Olive oil	Geographical origin	Mislabelling practices	Hierarchical Cluster Analysis (HCA) and Support Vector Machines (SVM)	To provide reliable information to customers and to ensure that the information on the label is followed.	[42]
			PLS-DA	To classify and predict four (4) commercial-grade virgin olive oils from Morocco	[42]
			PCA and PLS-DA	Geographical differentiation of a monovarietal olive oil	[44]
			PCA and (linear discriminant Analysis) LDA	Discrimination of Greek olive oil botanical origin	[45]

3.2 Presence of animal fats in vegetable edible oils

Edible oil adulteration is not only concerned with the blending of edible oil with other types of edible oils but also with replacing and substituting oil with other types of lipids such as mixing them with animal fats [46]. Thus, problems are rising due to this unethical behaviour. Animal fats that were mixed with edible oils have caused concern to certain groups of consumers. Owing to their religious duties, Muslims are forbidden from consuming any items containing lard or pork [47]. Thus, the detection of animal fats in edible oil is important to maintain the oils' qualities but can be complicated since some fats can have a similar composition to that of original oils. There were many emerging methods for detecting animal fats in edible oils such as using differential scanning calorimetry (DSC) [48], GC and HPLC to detect foreign fats in butter [49], portable electronic nose in the detection of lard [50] and usage of Near Infrared (NIR) spectroscopy in the detection of beef tallow in palm oil [51]. According to Fei [46], FTIR was a reliable and valid detection method since lipid consists of triglycerides and fatty acids which were well reflected in FTIR spectra mainly in region 3100-1700 cm^{-1} . Thus, such information can easily be obtained to identify lipids in certain species. The list of reviewed studies is in Table 3 below.

Al-Kahtani [52] investigated the presence of lard in a binary mixture of vegetable oils (corn, sunflower, palm, and olive oil) at different concentrations (1%, 5%, 10% and 20%) using an FTIR range of 4000-500 cm^{-1} and was measured in absorbance. The finding showed that there were only five (5) spectral regions that could differentiate between lard and vegetable oils which were 1405-1365 cm^{-1} , 1260-1198 cm^{-1} ,

935-910 cm⁻¹, 877-857 cm⁻¹ and 857-833 cm⁻¹. The authors later suggested using the chemometrics approach to further analyse and validate the results since the spectra of all samples appeared almost similar but with slight discrepancies. Hence, from the finding, FTIR managed to distinguish the presence of lard as small as 1% from the blends of vegetable oils by observing the differences in peaks of the spectral region.

Sim et al. (2018) [53] investigated the use of FTIR combined with partial least squares regression (PLSR) to estimate lard in palm olein oil. In the range of 4000-525 cm⁻¹, the spectra of lard, pure palm olein oil and contaminated olein oil (at 20% and 50%) have been obtained. It was said that the spectra patterns of pure and impure oil were comparable. Major absorption peaks were seen in the region of 3000-2800 cm⁻¹, 1700-1600 cm⁻¹ and 1500-900 cm⁻¹. The authors then used PLSR to predict lard contamination in palm olein oil by applying Fisher Weights, a multiclass variable selection method. Hence, there were five peaks (3006 cm⁻¹, 2852 cm⁻¹, 1117 cm⁻¹, 1236 cm⁻¹ and 1159 cm⁻¹) that were determined to be the most important discriminatory ability. Based on the five peaks except at region 3006 cm⁻¹, the intensity of the absorbance decreases as the concentration of lard increases, agreeing with the theory from Oyerinde and Bello (2016) [3], Gopal and Lakshmi Kantha (2017) [34] and [54] Antora et al. (2019). Contrast can be seen at region 3006 cm⁻¹ because lard is richer in cis C=CH bond compared to palm olein oil that was used in this study. As a result, the more abundant the bond, the higher the peak's strength. Authors mentioned that PLSR showed more consistent prediction using regions of 3006 cm⁻¹ and 1117 cm⁻¹ evaluated based on percentage root-mean-squares error (%RMSE) where %RMSE prediction between training samples (15.71) and test samples (16.03). Theoretically, a lower %RMSE in training samples indicates better prediction (Grace-Martin, n.d.).

FTIR is also commonly used with PLS in preventing fraudulent practices of foods. Munir [56] attempted to detect the presence of lard in various concentration ratios of the sunflower, canola, coconut, olive, and mustard oils. (edible: lard oil) of 10:0, 9:1, 7:3, 6:4, 4:6, 3:7,0:10. Munir [56] had selected 4000-400 cm⁻¹ to measure the spectra of the samples. Based on the spectral obtained in the region of 3100-400 cm⁻¹ contained certain shoulders and peaks representing specific functional groups and certain fatty acids. However, visually observed, all spectral patterns were similar except for some minor differences. This is due to the presence of common functional groups in all samples. However, the peak at region 3010 cm⁻¹ was absent only in the coconut oil spectrum. Meanwhile, only one peak can be observed at region 1100 cm⁻¹ in the coconut oil spectrum compared to other samples where two peaks can be seen in that region. For the lard spectrum, the peak position at region 1747 cm⁻¹ and peak shape at region 1650 cm⁻¹ was different than other edible oils. The other minor difference can also be seen in terms of absorbance intensities between all samples. Two specific regions 713.53-732.82 cm⁻¹ and 1246.75-1078.01 cm⁻¹. Only region 1246.75-1078.01 cm⁻¹ gave a good regression output compared to the first range. Based on the correlation coefficient (R²) of the region used was 0.9577 and the RMSEC of the PLS model was 0.0488 with a low root mean square error of prediction (RMSEP). In conclusion, FTIR coupled with PLS can be effectively employed in food certifying agencies or food departments for food

authentication from lard contamination. Continuous research is needed to ensure a continuous halal food supply that complies with shariah.

In summary, the detection of animal fats, particularly lard, in edible oils is a critical concern, especially for consumers with religious dietary restrictions. Various analytical methods have been explored to address this issue, with a particular focus on the application of FTIR. FTIR has proven to be a reliable tool for identifying the presence of lard in different edible oils, even at low concentrations, through distinctive spectral patterns.

Table 3. Uses of Fourier Transform Infrared Spectroscopy (FTIR) in Detection of Animal Fats in Edible Oils

Product	Issues	Sub-Issues	Chemometrics	Aim of study	References
Palm oil	Adulteration	Addition of lard	SLR, MLR and PLSR	Detection of lard in palm olein oil	[53]
Vegetable oils	Adulteration	Addition of lard	-	Detection of lard in corn, sunflower, palm, and olive oil	[52]
			PLS	Detection of lard contamination in five different edible oils: sunflower, canola, coconut, olive, and mustard oils	[56]

3.3 Edible oil mixed with other types of edible oil

To gain more profits, traders have commonly added other edible oils of lower commercial grade to another type of edible oil with higher commercial grade such as olive oil. Due to its distinct flavour and benefits, extra virgin olive oil (EVOO) has the highest quality and price in the oil and fat industry. Thus, EVOOs are more prone to adulteration. Since oils have a similar appearance, it is difficult to differentiate them without the need to use an advanced detection method. One of the methods that is commonly employed is FTIR since it provides rapid, fast detection and is non-destructive toward the sample.

Filoda [37] employed the uses of FTIR with PLS to identify EVOO from different edible oils (soybean, sunflower, corn, and canola oil) at different concentrations (1-80% (v/v)) rapidly. The total of adulterated samples was 68. All spectra were obtained between 3200-650 cm⁻¹ and were measured in absorbance. In addition, Xu [8] adulterated EVOO with peanut and rapeseed oil with the lowest concentration at 1.98% and the highest is at 51.77% randomly prepared and observed at spectra range of 4000-700 cm⁻¹.

Although the spectra pattern was similar, the authors found there were minor discrepancies between the spectra of samples in the form of peak or band intensity [4][8]. Saturated and unsaturated fatty acids, triacylglycerols, glycerol, sterols, tocopherols, and phenols were among the functional groups whose peaks were observed [8][57]. Major peaks for this

investigation can be seen at region 3080-2800 cm⁻¹. Small peaks at regions 3005 cm⁻¹ and 1654 cm⁻¹ signify the existence of unsaturated fatty acids agreeing with studies from Rohman [4]. Other observable regions were 1800 cm⁻¹, 1630 cm⁻¹, 1463 cm⁻¹, 1377 cm⁻¹, 1286 cm⁻¹, 1240 cm⁻¹, 1143 cm⁻¹, 1117 cm⁻¹, 1099-1098 cm⁻¹, 1039 cm⁻¹, 827 cm⁻¹ [4]. Some similarities and contradictions can be found when compared with [8] where the observable peaks were visible starting at 3005-2852 cm⁻¹ and 1743-721 cm⁻¹. Both studies mentioned that the lower the concentrations, the spectra were much more like that of EVOO. Ten variables (3042-2727 cm⁻¹, 2331-2253 cm⁻¹, 1936-1858 cm⁻¹ and 1304-910 cm⁻¹) which have the smallest cross-validation error between 0.16-0.27percent were selected for the calibration PLS model. Rohman [4] also used a similar range of the region as variables but based on the range capability to give the highest value of R² between actual and predicted and with the lowest error values. Meanwhile, Xu [8] also showed a positive and clear distinction between adulterated EVOO and non-adulterated EVOO using PCA with a 94.64% variance. They also used several multivariate calibrations such as the least-squares support vector machine (LS-SVM) where the results produced using this model were the most significant compared to others. Its optimum discrimination rates in preparation and prediction sets were 100% and 92.5 percent, respectively.

Another type of oil that has been adulterated and been studied is avocado oil. Like olive oil, avocado oil also comprises various health benefits and relatively high prices [58]. Common adulterants used are sunflower, soybean, corn, palm, and canola oil which are much cheaper compared to avocado oil. Rohman [59] has examined the adulteration of avocado oil with palm oil and canola oil. Meanwhile, Lumakso [6] and Jiménez-Sotelo [58] adulterate avocado oil with soybean, corn, and sunflower oil. All of them used FTIR presented in absorbance combined with PLS to validate their results. They measured the samples' spectra with a range of 4000-650 cm⁻¹ while Jiménez-Sotelo [58] used a range of 4000-550 cm⁻¹. Rohman [59] found several distinct peaks indicating differences between avocado oil with the adulterants at 3500-3250 cm⁻¹ and 1742-1715 cm⁻¹. These observations agreed with other findings from Lumakso [6] with a few more regions at 1099 cm⁻¹, 1114 cm⁻¹ and 975-900 cm⁻¹. While other major peaks are also similar in both studies conducted by them and Jiménez-Sotelo [58]. However, Jiménez-Sotelo [58] only presented the spectra of avocado oil with different concentrations (2-50%) of soybean-sunflower oils at regions 1600 and 550 cm⁻¹. The PLS analysis for all studies showed a good prediction capability of the model with R² values between 0.9961-0.9998 and low RMSEC and RMSEP values between 0.3963-0.86%. Thus, FTIR and PLS are an effective combination in detecting and quantifying the adulteration of avocado oil.

Other common oils that were adulterated were between relatively cheaper oils such as sesame, candlenut, coconut, sunflower, and rice bran oil as shown in Table 4. Different types of oils showed similar spectra patterns due to the same chemical composition which oil mainly consists of triglyceride and fatty acids. Therefore, the chemometrics method is needed to illustrate the differences between different types of edible oils. For example, Yuliani [60] used the principal component regression (PCR) and partial least square regression (PLSR) analysis tools to detect and quantify adulterants (sunflower, soybean, and corn oil) in candlenut oil. Mixed candlenut oil

and soybean oil were analysed at region 1765-1625 cm⁻¹ and 839-663 cm⁻¹ while mixed candlenut oil with corn oil was at 970-857 cm⁻¹. Furthermore, sunflower oil in candlenut oil was best quantified at region 3100-2800 cm⁻¹. The authors then obtained high R² with low RMSEC and RMSEP values. Thus, they concluded that the variables used influenced the discrimination of authentic candlenut oil from its adulterants.

In summary, the adulteration of high-quality edible oils, such as EVOO, with lower-grade oils is a common practice by traders seeking increased profits. This unethical behavior poses a challenge in differentiating oils based solely on visual inspection due to their similar appearances. FTIR with chemometrics emerges as a reliable and non-destructive method for rapid detection and analysis.

Table 4. Applications of Fourier Transform Infrared Spectroscopy (FTIR) in Edible Oils Adulteration with Different Types of Edible Oils

Product	Issues	Sub-Issues	Chemometrics	Aim of study	References
Olive oil	Adulteration	Addition of other vegetable oil	PLS	Detection of sunflower oil, corn oil, soybean oil and canola oil.	[37]
		Addition of peanut and rapeseed oil	PCR and PLS	Detection of grapeseed, soybean, and walnut oil	[4]
		Addition of peanut oil	LS-SVM	Detection of peanut oil and rapeseed oil in extra virgin olive oil	[8]
Avocado oil	Adulteration	Addition of other vegetable oil	PCR and PLS-R	Detection of peanut oil in extra virgin olive oil	[19]
		Addition of other vegetable oil	PLS	Detection of soybean oil, corn oil, palm oil, canola oil	[59] [6]
Virgin coconut oil	Adulteration	Addition of other vegetable oil	PLS	Detection of sunflower oil and soybean oil	[58]
		Addition of other vegetable oil	PCA	Detection of palm oil, mustard oil	[61]
Hempseed oil	Adulteration	Addition of other vegetable oil	PLS and FB-FiPLS	Detection of rapeseed oil, sunflower oil, sesame oil in hempseed oil	[56]
Sesame oil	Adulteration	Addition of other vegetable oil	PLS	Detection of hazelnut oil, canola oil, sunflower oil	[18]

Cand lenut oil	Adulteration	Addition of other vegetable oil	PCR and PLSR	Detection of candlenut adulterants	[60]
Vegetable oils	Adulteration	Addition of soybean and palm oil	PLSR	Detection of soybean oil and palm oil in sunflower oil and rice bran oil respectively	[54]

3.4 Edible cooking oil mixed with recycled cooking oil or gutter oil

Recent studies conducted by Lim [15] were to distinguish between fresh palm cooking oil with contaminated palm cooking oil and recycled cooking oil (RCO). Adulterated samples were made with a concentration of RCO ranging from 1-50% (v/v). The spectra analysis was performed using FTIR and was measured at the region of 4000-650 cm⁻¹. Again, both spectra were found to be similar in shape and peak position due to the presence of the major constituent, which was triglycerides. However, variations can be observed at regions 3529 cm⁻¹, 3442 cm⁻¹, 1655 cm⁻¹, 988 cm⁻¹, 964-968 cm⁻¹ and 908-904 cm⁻¹. Region 3529 cm⁻¹ and 3442 cm⁻¹ were said to be correlated with thermal degradation from continuous heating while others were due to the formation of trans double bond and reduction of a cis double bond. The PLS analysis resulted in an R² value of 0.998 while the RMSEC value was 0.796 and the RMSEP value was 3.218. Using DA, the fresh palm oil can be differentiated from the adulterated samples with 42.5% variations.

In addition, Kou [63] tried to adulterate several types of edible oils with recycled cooking oil at various concentrations of 0.1%-15% (w/w). The spectra were measured using a range of 6000-400 cm⁻¹ and region 1550-650 cm⁻¹ was used for detection assessment due to its evident peaks. However, no significant differences between pure edible oil, adulterated oils, and non-adulterated used frying oil except in terms of absorption intensities of the same peak. Hence, the authors concluded that adulterated oils could be distinguished by observing the peak position as well as the peak intensities. The qualitative analysis model established that the pure edible oils were separated from recycled frying oils and adulterated oils with a 100% recognition rate. Furthermore, the adulteration concentration can also be detected as low as 1% using the same analysis with an R² value of 0.9822 and a standard deviation of 0.2389. Both studies are listed in the following Table 5.

In summary, these studies underscore the efficacy of FTIR as a robust analytical tool for detecting the adulteration of cooking oils with recycled or contaminated counterparts. The ability to differentiate between pure and adulterated oils contributes to ensuring the quality and safety of edible cooking oils in the market.

Table 5. Applications of Fourier Transform Infrared Spectroscopy (FTIR) in Detection of Recycled Cooking Oil in Edible Oils

Product	Issues	Sub-Issues	Chemometrics	Aim of study	References
Palm oil	Adulteration	Addition of recycled cooking oil	PLS	Detection of recycled cooking oil in fresh palm oil	[15]
Vegetable oils	Adulteration	Used frying oils	PLS	Detection of used frying oils in rapeseed, soybean, peanut, sesame, virgin olive, pepper, sunflower, wild camellia, perilla seed, bitter apricot kernel, and mustard	[63]

4. CONCLUSION

Authentication of edible oils is essential due to ongoing consumer concerns regarding the quality of these products. Due to its demand in the market, other unethical sellers, traders, and manufacturers have associated pure edible oils with different types of oils to gain more profits. However, these fraudulent practices might lower the quality and benefits of edible oils. Therefore, many have chosen advanced analytical methods such as FTIR since they are fast, accurate, require minimal sample preparation, and are non-destructive to the sample. Based on the literature, most edible oils showed a similar spectra pattern due to their chemical composition, which is mainly triglycerides. The combination of FTIR with chemometrics analysis has resulted in more thorough and better results. The popularity of FTIR has shown that this method is suitable for authenticating not only oils and fats but also any types of food that require the attention of analytical methods. With the advancement of analytical methods, the food industry can be assured of safety, as these developments will help detect and prevent the presence of any adulterants.

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