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Edible Oil Discrimination by Fourier Transform Infrared (FTIR) Spectroscopy and Chemometrics

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ABSTRACT

The advancements in Fourier transform infrared (FTIR) spectroscopy have expanded applications in food science, including in the analysis of edible oils and fats. Infrared spectroscopy is increasingly being employed to track changes in edible oils, determine these changes, and detect unauthorized modifications. This article presents a review of some of the most significant applications of infrared spectroscopy for distinguishing edible oils from 2015 to 2022.

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Fourier transform infrared (FTIR) spectroscopy; partial least squares–discriminant analysis (PLS-DA); principal component analysis (PCA); edible oil analysis

Introduction

The term edible oil refers to fatty liquids obtained from vegetables, animal tissues, or microorganisms that are suitable for human consumption. Although both fats and oils contain fatty acids/triacylglycerols (TAG) as the main constituents, their primary distinction is that at room temperature, the physical state of fats is solid while oils exist as liquids (Bunaciu, Aboul-Enein, and Hoang 2020).

Vegetable oils hold a position of great importance among agricultural products globally, offering a diverse range of products that can provide essential contributions to the diet. For example, it is essential for adults to consume approximately 5 g of linolenic and unsaturated fatty acids daily since the human body cannot produce them (Pitts, Dorling, and Pattie 2007).

The domestication of sheep, goats, and cattle occurred approximately 10–11,000 years ago, while the cultivation of olive oil dates back roughly 6,000 years. Compared to these traditional sources, the use of lipids from dairy sources and refined vegetable oils is a relatively new phenomenon (Cordain et al. 2005). Throughout history, people have used these oils in different ways, ranging from religious ceremonies to cooking, frying, or in food product formulations (Issaoui and Dekgado 2019).

It is well-known that the majority of edible oils are obtained from seeds, with only olive, coconut, and palm oils being derived from fruits. Nowadays, the most frequently

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used edible oils are soybean, olive, sunflower, palm, coconut, and rapeseed oils (Li et al. 2019).

The role of food in human nutrition is critical, and any factor that undermines its safety and quality can have significant health and economic consequences for society. Given the intricacy of food, it is crucial to employ precise and accurate techniques for assessing its quality and authenticity.

Food fraud has been prevalent since ancient times. One common food fraud involves adding low-value or non-existent substances to expensive or valuable products, motivated by the desire for monetary gain. Another motive is to conceal the unpleasant flavor and appearance of decaying food, which was typically achieved by adding coloring and flavoring agents (Bush 2002).

Adulteration has become increasingly sophisticated over time, often in response to or in anticipation of developments in analytical sciences. Recently, concerns have arisen due to specific types of fraud involving the geographic origin of ingredients and finished goods (Spink and Moyer 2011; Oliveri and Downey 2012; Danezis et al. 2016; Tahir et al. 2022). The application of chemometrics methods can help handle large quantities of data in the interpretation of complex patterns and relationships, allowing for discrimination between authentic and adulterated products (Valand et al. 2020; Borras et al. 2015; Ye and Meng 2022).

Edible oils are susceptible to adulteration due to chronic shortages and price fluctuation. This is an important issue because adulteration can have detrimental effects on consumers' health (Yadav 2018). Addition of argemone oil (AO) to edible oils has been associated with glaucoma, loss of eyesight and epidemic dropsy due to the presence of alkaloids such as dihydrosanguinarine and sanguinarine in argemone (Das and Khanna 1997). Furthermore, the adulteration of mustard oil with AO and butter yellow may cause gallbladder cancer (Mishra et al. 2012). For oil identification and adulteration detection, several methods have been utilized to measure the refractive index, iodine value, hydroxyl value, and saponification value as well as to determine the composition. Advanced techniques, such as nuclear magnetic resonance (NMR), Fourier-transform infrared (FTIR) spectroscopy, Raman spectroscopy, and mass spectrometry (MS), have been employed for food authentication in recent years. These techniques provide high-resolution data in conjunction with chemometrics to distinguish authentic and adulterated edible oils (Valand et al. 2020; Rohman 2017a; Kettner et al. 2010; Berrueta, Alonso-Salces, and Héberger 2007; Rohman 2017b; Mohammed et al. 2021; Rohman et al. 2021).

According to the literature, there has been the growing interest in the development of nondestructive, accurate, and inexpensive methods for edible oil authentication. Thus, the aim of this paper is to provide an overview on the application of FTIR spectroscopy from 2015 to 2022 for the differentiation of edible oils, with emphasis on the type examined and the geographic location.

Edible oil discrimination

Argan oil, derived from the *Argania spinosa* L., is highly valued for its dietary, therapeutic, and dermatologic benefits. It is particularly valuable to the southwestern

Moroccan populations and has been incorporated into the European diet since 1998. However, since argan trees grow in diverse geographical areas, the detailed chemical composition of argan oil has not yet been fully established. Researchers analyzed the chemical composition of markers including free acidity, peroxide value, spectrophotometric indices, fatty acids, tocopherols, and sterols from five regions to classify protected geographic indication argan oils using FTIR fingerprinting and chemometrics (Figure 1) (Kharbach et al. 2017).

The spectra are characterized by the symmetrical and asymmetrical stretching of $-CH_2$ at 2924 and 2852 cm^{-1}, C = O stretching at 1743 cm^{-1}, CH_2 and CH_3 scissoring at 1463 and 1377 cm^{-1}, and –C–O stretching at 1238, 1163, 1114 and 1099 cm^{-1}. Additionally, the CH_2 rocking peak was observed at 721 cm^{-1} which is consistent with previous studies (Sinelli et al. 2010; Kharbach et al. 2021). Principal component analysis (PCA) and partial least squares–discriminant analysis (PLS-DA) models were successful in accurately classifying the samples according to their geographic origin.

A novel analysis combining near-infrared spectroscopy (NIRS) and chemometric tools was developed to detect adulteration of argan oil by low cost vegetable oils. This technique detected adulteration levels as low as 0.35% (w/w) in less than 1 s (Farres et al. 2019). PCA models were developed for two spectral ranges, 500–1000 nm and 1000–1700 nm, with the latter producing better results. Partial least squares (PLS) yielded the best results for the 500–1000 nm region. Support vector machine (SVM) was also employed for regression for mid-IR spectra for detecting commercial products in argan oil (El Orche et al. 2020).

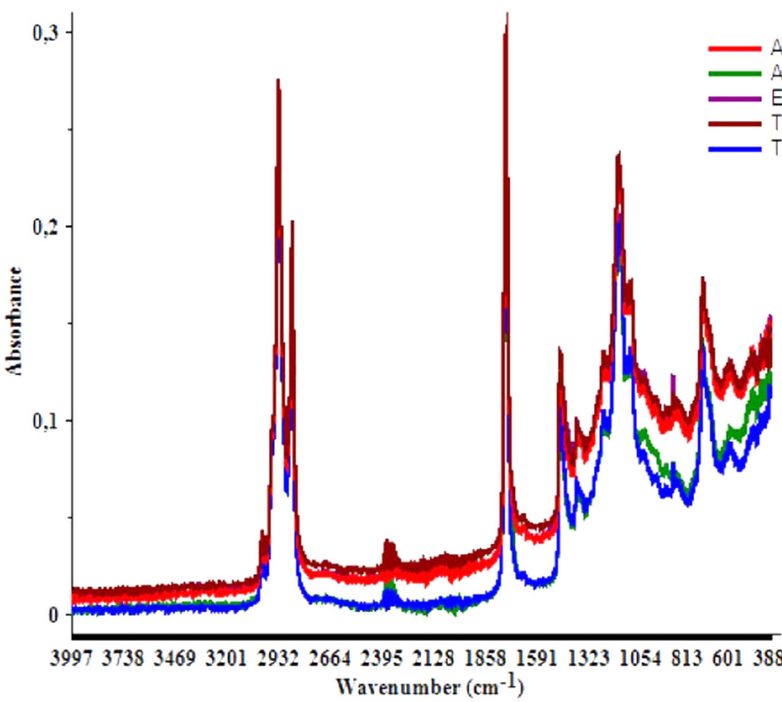

Figure 1. Infrared spectra of argan oils from Morroco. Locations: Agadir – A, Ait-Baha – A-B, Essaouira – E, Taroudant – T and Tiznit – Tiz. Reproduced with permission from Kharbach et al. (2017).

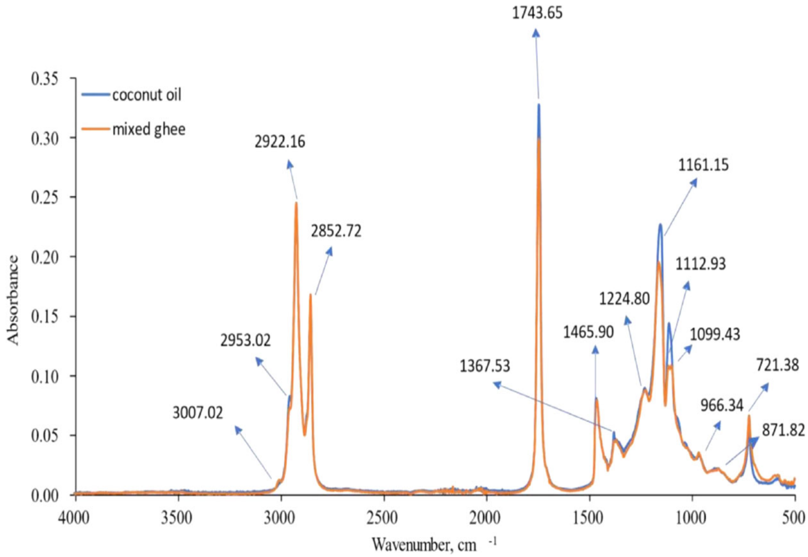

Figure 2. Infrared spectra of pure mixed ghee and coconut oil. Reproduced with permission from Gandhi et al. (2022).

Coconut oil has a high concentration of saturated fat, and it is believed that consuming this food may increase the risk of developing cardiovascular diseases. As a result, the consumption of coconut oil has decreased significantly in recent years.

Virgin coconut oil (VCO) is considered to be a functional food oil due to its beneficial properties (Marina, Che Man, and Amin 2009). One of the key indicators of VCO quality is the level of free fatty acids, which are produced by the hydrolytic rancidity of triglycerides in vegetable oils (Osawa, Goncalves, and Ragazzi 2007). To determine the concentration of free fatty acids in VCO, PLS was performed using FTIR with attenuated total reflectance (ATR) (Marina, Wan Rosli, and Noorhidayah 2015). In a recent study, a rapid methodology was reported for detecting the authenticity and adulteration of virgin coconut oil by ATR-FTIR spectroscopy in combination with data-driven soft independent modeling of class analogy (DD-SIMCA) (Neves and Poppi 2020). The developed DD-SIMCA models were highly sensitive (from 88.23% to 100%) and specific (95.74% to 100%) for the presence of canola, corn, sunflower, and soybean oils as adulterants in virgin coconut oil.

Ghee, which is also referred to as anhydrous milk fat, is a form of clarified butter. It is made by heating butter to remove moisture and caramelized particles, resulting in a product that consists solely of milk fat. It is widely used in India and is known for its health benefits, including its ability to slow down aging and assist the immune system as well as to help with certain eye diseases. Compared to other oils and fats, it is considered to be highly valued and often demands a premium price. This has led to the addition of adulterants to ghee. One common adulterant is coconut oil, which may be added from 2% to 15%. The concentration of coconut oil in pure ghee was determined using ATR-FTIR with PCA, SIMCA, PLS, and principal component regression (PCR) (Gandhi et al. 2022).

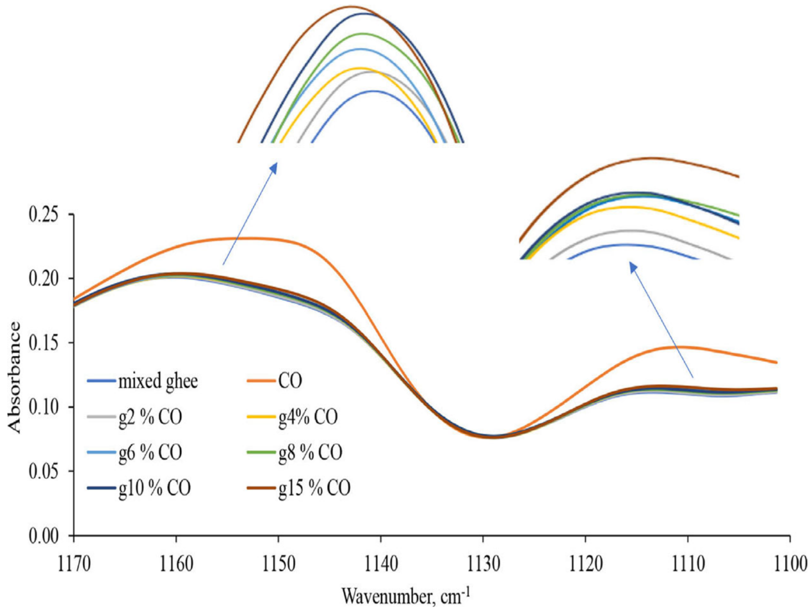

Figure 3. Infrared spectra of pure ghee (g) and ghee adulterated by coconut oil (CO). Reproduced with permission from Gandhi et al. (2022).

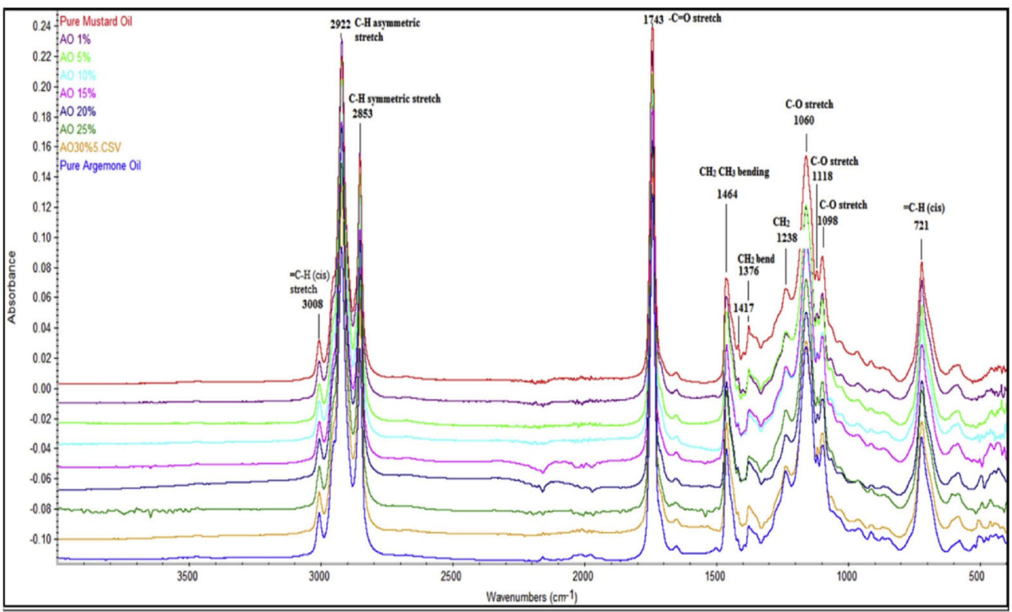

Figure 4. ATR-FTIR spectrum of mustard oil (MO) and mustard oil adulterated with argemine oil (AO). Reproduced with permission from Jamwal, Kumari, Balan, et al. (2020).

Figure 2 illustrates a comparison of the FTIR spectra from pure coconut oil and ghee mixed with coconut oil. Figure 3 presents a more detailed view of the changes in the spectra observed when ghee was adulterated with coconut oil.

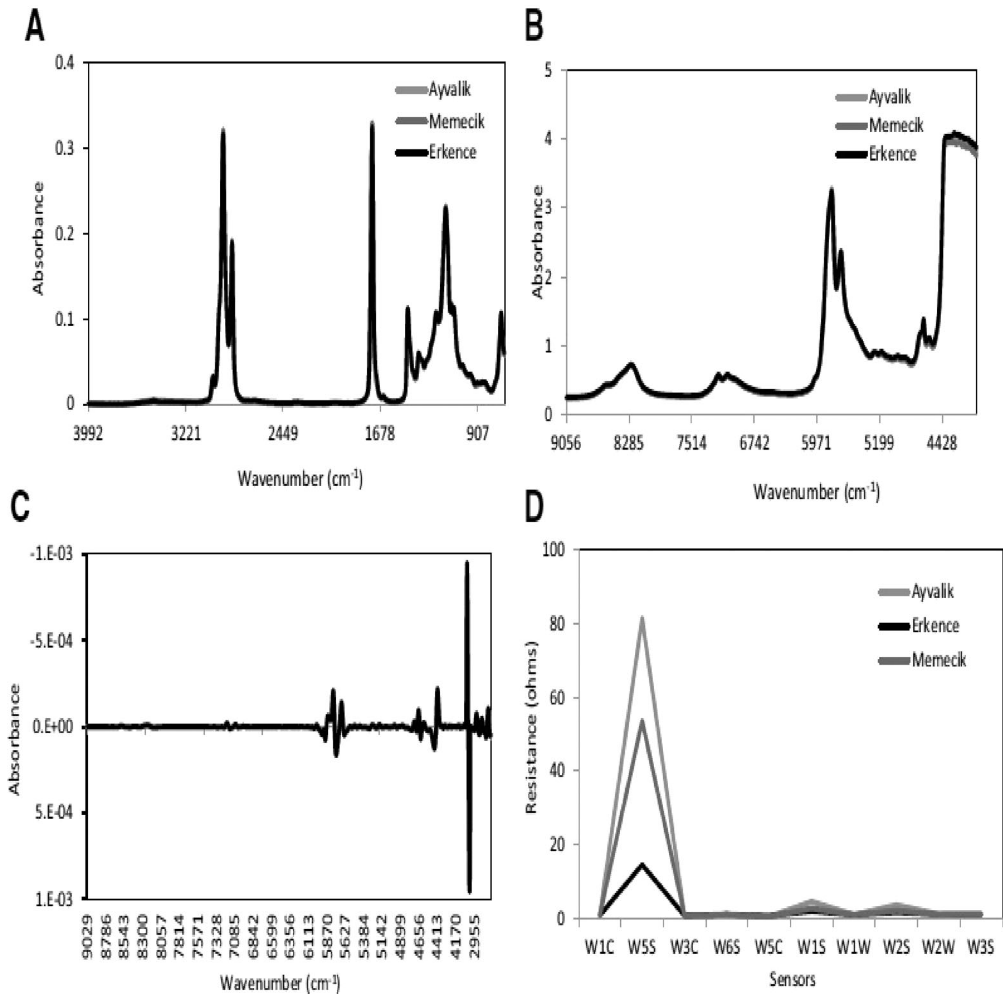

Figure 5. Characterization of olive oils from Ayvalik, Erkence, and Memecik, Turkey by (a) FT-MIR, (b) FT-NIR, (c) standard normal variate and second-order derivative filtered FT-NIR-MIR, and (d) e-nose measurements. Reproduced with permission from Jolayemi et al. (2017).

Similar spectra for pure ghee (Antony et al. 2018; Upadhyay, Jaiswal, and Jha 2018) and coconut oil (Gervajio, Withana-Gamage, and Sivakumar 2020) have been reported. The functional group regions were nearly identical, but slight variations in the fingerprint regions of the two oils were detected.

Mustard oil (MO) is a basic ingredient in the Indian diet due to its health advantages, such as boosting the good cholesterol ratio and providing omega-3 and omega-6 fatty acids (Das and Khanna 1997; Mishra et al. 2012). However, MO is frequently adulterated with argemone oil, butter, palm oil, and linseed oil. A study was conducted to use ATR-FTIR in conjunction with chemometrics, PCA, and linear discrimination analysis (LDA) to evaluate the adulteration of argemone oil (AO) in MO (Jamwal, Kumari, Balan, et al. 2020; Jamwal, Kumari, Balan, et al. 2021). PCR and PLS-R were compared using normal, first, and second derivative spectra. The mid-infrared spectra between 4000 and 400 cm^{-1} were collected for pure MO and adulterated MO (Figure 4).

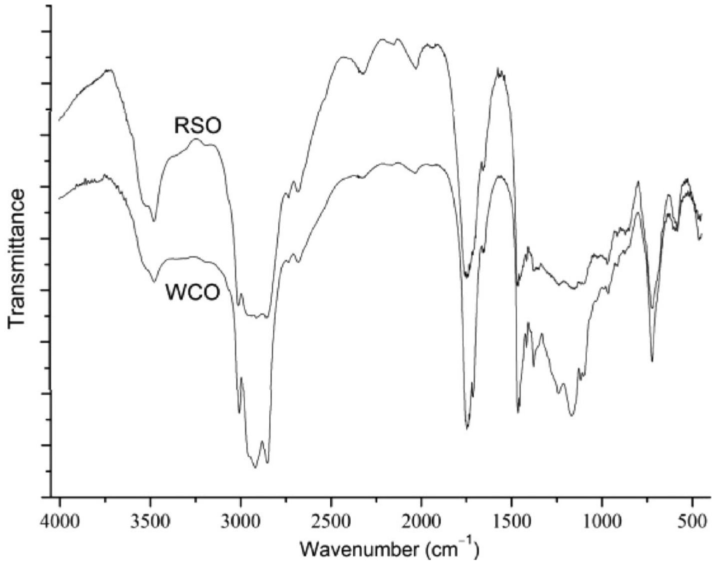

Figure 6. Infrared spectra of rapeseed oil (RSO) and rapeseed oil with waste cooking oil (WCO). Reproduced with permission from Wu et al. (2015).

Similarities exist in the composition of oil samples, which can make it challenging to distinguish the peaks. All plant oils have similar composition, with triacylglycerol being the primary component, accounting for 92%, followed by low concentrations of di- and mono-acylglycerols (5%), and minor levels of other components. However, the classification and quantification of AO in MO was reliably performed with a detection limit of 1%.

Extra-virgin olive oil (EVOO) is a crucial element of the Mediterranean diet and obtained from fresh fruit using mechanical techniques (Li and Wang 2018). It is a well-studied due to its distinctive sensory properties, high nutritional value, and established health benefits. During storage, these characteristics may change (Jolayemi et al. 2017).

In an effort to differentiate olive oil samples from Ayvalik (A), Memecik (M), and Erkence (E) in western Turkey between 2012 and 2015, an investigation was performed employing FT-NIR (Fourier transform near infrared), FT-MIR, and an electronic nose (e-nose). To analyze the results, orthogonal partial least squares–discriminant analysis (OPLS-DA) was used (Jolayemi et al. 2017). Additionally, standard normal variate (SNV) and second-order derivative filtering were applied to all spectra. Figure 5 shows representative spectra of the olive oils. Accurate classification of the samples was achieved using FT-NIR and the combination of FT-MIR-NIR spectra. Although the e-nose had relatively low discriminative ability, it may be a useful supplement to subjective sensory analysis by humans. The characteristic peaks in the spectra were previously identified (Guillen and Cabo 1997; Guillen and Cabo 2002).

Rapeseed oil (RSO) is most important oilseed in China. Nonetheless, waste cooking oil (WCO), a low-cost and widely available material, has been frequently used to adulterate RSO, posing a serious problem. FTIR with PCA, PLSR, and support vector machine (SVM) were investigated to quantify adulteration in RSO (Li et al. 2019). PLSR was the most effective with a limit of detection of 2%. In another study, FTIR

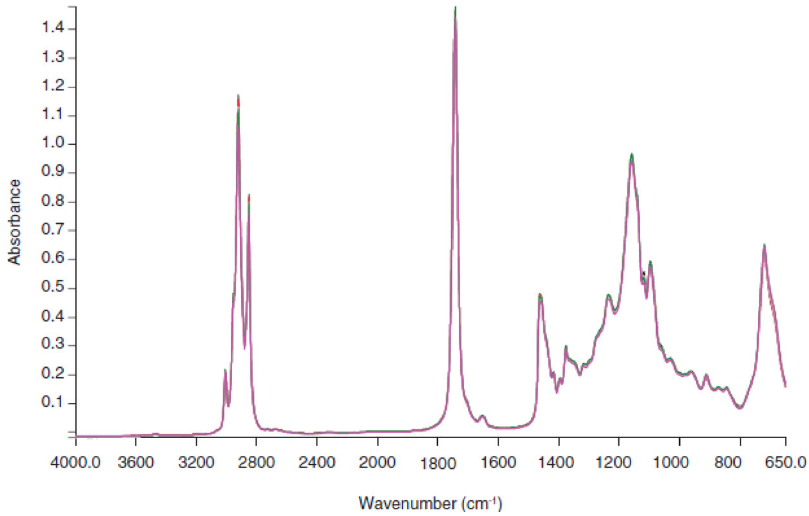

Figure 7. Infrared spectra of sunflower oil and sunflower oil adulterated at 60%. Reproduced with permission from Uncu, Ozen, and Tokatli (2019).

spectroscopy was used to quantify WCO as adulterants in selected RSO samples with multivariate calibration (PLS and PCR) (Wu, Li, and Tu 2015). The spectra for RSO and WCO from 4000 to 450 cm^{-1} are shown in Figure 6 (Guillen and Cabo 1997).

Sunflower seeds have high oil content and are an important crop. Uncu and colleagues utilized FTIR spectroscopy and chemometric tools to characterize the adulteration of sunflower oil with safflower oil and compared the results to the fatty acid profile (Uncu, Ozen, and Tokatli 2019). Mid-IR spectra of pure sunflower and safflower oils, as well as their mixtures, were collected for authentication, as shown in Figure 7.

The differences between pure sunflower oil and the adulterated oil were minimal and not readily distinguishable to the naked eye. The spectra were analyzed by orthogonal partial least squares–discriminant analysis (OPLS-DA) and SIMCA. The second derivative of the spectra (4000 to 650 cm^{-1}) was used to remove noise and baseline shifts providing a dataset of 3351 variables that identified adulteration at concentrations as low as 10%.

Conclusions

In recent years, vibrational spectroscopy has been employed for the quality control of fats and oils due to the distinct fingerprint of the spectra. FTIR is well established for laboratory and industrial use and allows the characterization of oils, allowing the identification of adulteration. This technique provides information about the composition, geographical origin, processing, and storage. For the food industry to derive maximum benefit, advancements should prioritize the development of better algorithms, faster integration times, and handheld devices. It is anticipated that portable and intelligent instrumentation will further improve the evaluation of the genuineness and the provenance of these foods.

Authors' contribution

Andrei A. Bunaciu contributed to conceptualization and writing the original draft. Vu D. Huang contributed to data curation, supervision, and writing - reviewing and editing, Hassan Y. Aboul-Enein contributed to data curation, supervision, and writing - reviewing and editing.

Disclosure statement

The authors declare no conflicts of interest.

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