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REVIEW ARTICLE

Raman Spectroscopy for Edible Oil Authentication: A Review

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Abstract

There have been many prior studies on oil authentication, especially edible oils owing to the need for genuine edible oils on the market and consumers. Therefore, analytical approaches have been one of the detection and identification methods for adulteration of edible oils. The authentication of edible oils using Raman spectroscopy is a common vibrational spectroscopies analysis technique. Raman spectroscopy has shown some advantages such as rapid, non-invasive analysis and no sample pre-treatment providing ease to research. Hence, the purpose of this paper was to discuss the previous studies on edible oil authentication using an analytical approach, particularly Raman spectroscopy and its applications that have been used for oil authentication analysis. Edible oil authentication and adulteration are important to differentiate genuine edible oil from fake ones and to prevent fraud. This paper also discussed on analysis of qualitative and quantitative edible oils authentication, along with the cases of adulteration analysis in edible oils such as the determination of foreign contents in edible oils, quality analysis of genuine edible oils, identification of various types of vegetable oils, adulterated oils in food products and characterization of waste cooking oils.

Keywords: Raman spectroscopy, Raman spectra, Edible oil, Authentication, Adulteration

Introduction

This Raman spectroscopy is an analytical technique that was adopted for oil authentication or oil purification. It is a highly sensitive technique that yields individual molecular spectra or fingerprints (Lv et al., 2016). Thus, it helps to analyse some modes in a system such as vibration, rotational and other low-frequency where this relies on Raman scattering which is the inelastic scattering of monochromatic light that interacts with a matter (Oroian & Ropciuc, 2017). In the 1970s, it was reported that the early work on Raman scattering had results in non-destructive analysis, elevated

sensitivity and single molecule detection that helps in the improvement of Raman signal when pyridine molecules in the rough surface of the silver electrode are used (Fleischman et al., 1974). Over the years, Raman techniques managed to overcome the constraints of traditional methods and have been able to be an important analytical tool for assessing food quality (Yang et al., 2019).

Raman technique is vibrational spectroscopy where the combination of Raman spectroscopy with chemometrics are useful techniques for determining the authenticity, especially for Extra Virgin Olive Oil (EVOO) despite it has infrared and near-infrared. This technique relies on the variations of Raman band intensity used for the evaluation of edible oils quality and has some advantages such as rapid, non-invasive technique, no sample pre-treatment and the low detection of limit to evaluate the quality of edible oils (S. Duraipandian et al., 2019; Philippidis et al., 2016). In addition, a small volume of sample is required for Raman analysis (Oroian & Ropciuc, 2017). Hence, mapping and display, discriminant and clustering methods are being used for pattern recognition (Kwofie et al., 2020).

For these benefits, Raman spectroscopy has been used as one of the methods in the detection and identification of edible oil adulteration. Genuine edible oils are highly demanded on the market has resulted in some serious issues that need to be concern such issues edible oil adulteration may be the most prevalent concern on the market such as fallacious acts on the vegetable oils to quality, the use of waste cooking oils in restaurants, the presence of foreign substances and even the adulteration of vegetable oil with animal oil. These issues may pose risks to human health including the concerns about the use of allergy or prohibited components in edible oils based on their diets. Therefore, Raman spectroscopy has been used along with the combination of multivariate statistics in the authentication of edible oils analysis (S. Duraipandian et al., 2019).

There were some examples from the previous studies in the analysis of oil authentication using Raman spectroscopy such as in the utilization to characterize macadamia and pecan nut oils with other oil, cheaper seed oils such as corn and sunflower oils (Carmona et al., 2015). The differentiation between vegetable oils and other cheaper oils is based on their fatty acid profile by using Raman spectroscopy. Furthermore, it has shown to be an effective method to obtain qualitative and quantitative information, especially for olive oils of Andalusian extra virgin olive oil (EVOO) where the determination of qualitative includes harvest year, olive variety, and geographical origin whereas quantitative determination includes the degree of unsaturation of fatty acids (Sanchez-Lopez et al., 2016). Moreover, EVOO is a lipophilic product that consists of lipids mainly mono-unsaturated fatty acids followed by poly-unsaturated fatty acids and free fatty acids is catalysed by lipase during the storage and transportation process, (Bajoub et al., 2018; Jimenez-Lopez et al., 2020).

Moreover, there was also a combination of Raman spectroscopy with chemometrics tools for the analysis of the edible oils' authentication and adulteration. For instance, Greek (Cretan) adulterated with sunflower oil is used by using a combination method of Raman spectroscopy and chemometrics and it has shown that Raman spectroscopy studied the concentration analysis of olive oil adulteration (Philippidis et al., 2016).

In addition, a comparable method shows the effective and reliable compositional analysis and authentication for the characterization of edible oils/ghee and spread (Ali et al., 2016). Hence, the use of Raman spectroscopy and its application to authenticate edible oils were discussed in this paper according to previous studies that have been conducted. There was a further discussion of qualitative and quantitative analysis of edible oils authentication, along with the adulteration analysis cases in edible oils such as the determination of foreign contents in edible oils, quality analysis of genuine edible oils, identification of different types of vegetable oils, adulteration of food products with oils and characterization of waste cooking oils.

Authentication of Edible Oils

There are various types of edible oils authenticated using a Raman spectroscopy that was focused on the fatty acid profile in edible oil to investigate other parameters including the harvest year, variety, geographical origin or Protected Designations of Origin (PDO), maturation stages of cultivars, cultivar discrimination, agro-climatic variables, physiochemical characteristics, iodine, acidic peroxide and saponification values.

Besides, the determination of the content of bioactive components in edible oils is also one of the methods for the authentication of oils. Authentication is important to produce a high standard quality of oil despite to recognize fake oils (Rocha et al., 2016).

The relationship between fatty acid profile and other parameters in edible oils

The studies on edible oils were mostly focused on their content of fatty acids especially on the degree of unsaturation. Edible oil is a mixture of triglycerides that differ in their relative fatty acids composition (Kwofie et al., 2020). Vegetable oil quality was evaluated based on its content of unsaturated fatty acids. Lv et al. (2016) and Qiu et al. (2019) both experimented with the evaluation of vegetable oil quality according to the unsaturated fatty acid contents analysis using Raman spectroscopy. According to Lv et al. (2016), the unsaturated fatty acids with C=C bonds include oleic acid, linoleic acid and α-linolenic acid correlated to integrated surfaced-enhanced Raman scattering (SERS) intensity peak at 1656 cm^{-1} (S¬1656). It was reported that when the unsaturated fatty acids content is increased, the stronger the intensity bands at 1656 cm-1. To summarize, the SERS signal became stronger result of the increase in the C=C bond concentration.

Thus, the authenticity of peanut oil, sesame and soybean oils was evaluated according to the National Standards of China by converting unsaturated fatty acids into the equivalent total content of oleic acid to proceed with the analysis of the box plot of pure oleic acid S1656 value. Gas chromatography–mass spectrometry (GC-MS) analysis is used to verify the accuracy of this method and it shows the relative errors of this method is less than 5%. In addition, Qiu et al. (2019) reported that the oleic acid, linoleic acid and α-linolenic acid intensity ratios of Raman spectroscopy analyses between 1655 cm^{-1} to 1440 cm^{-1} and 1265 cm^{-1} to 1300 cm^{-1} enable the use of qualitative comparison in edible vegetable oils for polyunsaturated fatty acid and monounsaturated fatty acid contents with similar total unsaturated fatty acid content. Meanwhile, an intensity ratio of 1655 cm^{-1} to 1440 cm^{-1} could be obtained using two-dimensional correlation spectroscopy (2DCOS), thus it helps in the differentiation of the types of polyunsaturated fatty acids in edible oils. Moreover, to distinguish between waste cooking oil and vegetable oils, Huang et al. (2016) applied the intensity ratio of 1654 cm^{-1} to 1438 cm^{-1} using Raman spectroscopy and partial least squares – discriminant analysis (PLS-DA) according to their unsaturation degrees. This study reported that the waste cooking oil is the only sample that is located below the line of y=0.55 meanwhile olive, peanut and corn oil were located above the line in the classification map.

There were also studies using Raman spectroscopy to determine qualitative and quantitative information in edible oils such as unsaturated fatty acid contents, harvest year, variety, geographical origin or PDO and so on. Raman spectroscopy has produced the lowest errors in fit and prediction when with the partial least squares (PLS) for the estimation of fatty acid composition for monounsaturated fatty acids in Andalusian EVOO compared to saturated and polyunsaturated fatty acids (Sánchez-López et al., 2016).

The study also reported that discrimination analysis (DA) was successfully differentiated among the harvest year, olive variety, geographical origin and PDO with the highest percentages of 94.3%, 84.0%, 89.0% and 86.6%, respectively. Gouvinhas et al. (2015) also reported that Raman spectral data using principal component regression (PCR) and partial least squares regression (PLS-R) could produce good calibration and prediction values with coefficient determination of higher than 0.933 to monitor the free acidity and peroxide values in EVOOs. In

addition, Raman spectroscopy has used the principal of the component analysis (PCA) and linear discriminant analysis (LDA) results in the classification of EVOOs from the same experimental orchard according to the cultivar at about 94.4% and EVOO's quality was assessed based on the ratio of lutein/β-carotene content estimation (Portarena et al., 2019). Detection of high linear correlations between the fatty acid profile of EVOO with the harvest and agro-climatic variables based on the PCA of Raman spectra was also identified (Sánchez-Rodríguez et al., 2019). Based on Raman spectra, the unripe kernel oil produced higher values of unsaturated fatty acids at 3005 cm^{-1} compared to ripe kernel oil, Souza et al. (2019) reported.

A study found a good correlation between the integral intensity ratio of Raman spectral data at 1650 cm^{-1} for v(C=C) and 2850 cm^{-1} for v(CH2) vibrations with iodine values to discriminate thirteen different edible oils (Dymińska et al., 2017). Thus, the combination of Raman spectroscopy and other various methods successfully assessed the parameters that could help in authenticating different types of edible oils. **Table 1** summarized the studies on the relationship between fatty acid profile and other parameters in edible oils.

Table 1. Relationship between fatty acid profile and other parameters in edible oils

Product	Issue	Sub-Issue	Aim of the Study	Uses of Technique	References
Peanut oil, sesame oil, and soybean oil	Authentication	Indication of unsaturated fatty acid contents in vegetable oils	Authentication of vegetable oils based on the unsaturated fatty acid content which conforms to certain quality control specifications such as National Standards of China	SERS and GC-MS	Lv et al. (2016)
Vegetable oils	Authentication	Unsaturated fatty acid analysis	Quality evaluation of edible vegetable oil based on the content of unsaturated fatty acid	Raman spectroscopy with 2DCOS and relative intensity ratio	Qiu et al. (2019)
Olive oil, peanut oil, corn oil and waste cooking oil	Authentication	Unsaturated fatty acid content	Identification of waste cooking oil and qualified edible vegetable oil	Raman spectroscopy and PLS-DA	Huang et al. (2016)
EVOO	Authentication	Fatty acid contents, harvest year, olive variety, geographical origin and Andalusian PDO	Determination of qualitative (harvest year, olive variety, geographical origin or Andalusian PDO) and quantitative information (fatty acid contents) of Andalusian EVOO	FT-Raman spectroscopy and GC-FID with PLS and DA	Sánchez-López et al. (2016)
Olive oil	Authentication	Origin and maturation stages of cultivars	Discrimination of varietal origin of olive oils and different maturation stages of cultivars	Raman spectroscopy with PCA, DA, PCR and PLSR	Gouvinhas et al. (2015)
EVOO	Authentication	Cultivar discrimination, fatty acid profile and	Investigation of fatty acid and carotenoid content in connection	Raman spectroscopy and	Portarena et al. (2019)

		carotenoid characterization	with the cultivar and the olive maturation stage.	GC-FID with PCA, LDA and PLS-R	
EVOO	Authentication	Fatty acid profile, harvesting and agro-climatic variables	Determination of fatty acid profile of EVOO based on harvesting and agro-climatic variables	Raman spectroscopy and GC with PCA	Sánchez-Rodríguez et al. (2019)
Kernel oil	Authentication	Fatty acid composition, physiochemical characteristics and oxidative stability	Assessment of the oil quality from kernels by observing the ripening time, fatty acid composition, physiochemical characteristics and oxidative stability	GC-FID, ^{1}H NMR and FT-Raman with index calculation	Souza et al. (2019)
Edible oils	Authentication	Fatty acid composition, iodine, acidic peroxide and saponification values	Discrimination analysis of different oils	FTIR and FTRS with PCA	Dymińska et al. (2017)
Fish oil	Authentication	Polyunsaturated fatty acid, ethyl esters and oxidation	Quantification of polyunsaturated fatty acid, detection of esthyl esters and oxidation	Raman spectroscopy	Killeen et al. (2017)

Bioactive Component Contents in Oils

Bioactive component content in edible oils is studied using Raman spectroscopy. SERS is a rapid, nondestructive, and reliable analytical technique (Camerlingo et al., 2019). A study by Broadhurst et al., (2018) reported that Raman spectroscopy could detect the thermal gradient of lipids since the increase in Raman intensity, line broadening and frequency shifts were correlated with the melting of lipids in fish oils.

This study demonstrated that docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA) in five fish oils were studied based on Raman data and band assignments to differentiate and authenticate between the commercial fish oils. Additionally, Raman spectroscopy successfully determined good Deterioration of Bleachability Index (DOBI) and carotenoid contents in crude palm oil (Nokkaew et al., 2019). It was determined about 1000 cm^{-1} and 1500 cm^{-1} of carotenoid content could help in the quality of crude palm oil determination.

In another study, carotene which is a compound of carotenoid in EVOOs had been characterized using SERS at 1150 cm^{-1} and 1525 cm^{-1} together with phenol at 1237 cm^{-1} and oleic acid at 1350 cm^{-1} (Camelingo et al., 2019). Moreover, bioactive components in essential oils could also be quantified by using Raman spectroscopy (Lafhal et al., 2015). Based on previous studies described, it could aid in investigating the bioactive component contents in edible oils as well as differentiate between each edible oil.

The prediction of quantification on main compounds in the essential oils especially on species such as lavender, lavender and their varieties with high accuracy allows the discrimination and authenticity detection of the lavender. Hence, this study could also help in detecting the bioactive components in other edible oils to determine their quality. **Table 2** shows studies on bioactive component content in oils.

Table 2. Bioactive component contents in oils

Product	Issue	Sub-Issue	Aim of the Study	Uses of Technique	References
Fish oils	Authentication	Docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA) contents	Identification of DHA and EPA sources in commercial fish oils	GTRS, DSC, and Raman spectroscopy	Broadhust et al. (2018)
Palm oil	Authentication	Carotenoids and deterioration of bleachability index (DOBI) content	Determination of DOBI and carotene content in the crude palm oil	Raman and FT-NIR spectroscopy with PLS	Nokkaew et al. (2019)
EVOO	Authentication	Contents of carotene, oleic acid and phenols	Identification of bioactive components in EVOO such as carotene, oleic acid and phenols	SERS with DWT	Carmelingo et al. (2019)
EVOO	Authentication	Phenolic content	Identification of phenolic content	HPLC-MS, FAST HPLC	
Lavandin oil and lavender oil	Authentication	Terpenoid compound contents	Quantification of terpenoid compounds in essential oils	GC and Raman spectroscopy with PCA, PLS and PLS-DA	Lafhal et al. (2015)
Cold pressed oil	Authentication	Fatty acids, tocopherols, polyphenols, sterols, and the total phenol contents	Identification of bioactive compounds and total phenol contents of cold pressed oils from safflower and camelina seeds	HPLC analysis	Presti et al. (2017)
Carlina acanthifolia subsp. utzka root oil	Authentication	-	Identification of chemical stability and bioactive compound	Raman spectroscopy, ATR-IR and NMR spectroscopy	Strzemski et al. (2017)

Hemp seed oil	Authentication	Poly-unsaturated fatty acids (PUFAs)	Determination of chemical composition and quality paramaters	UHPLC with quadrupole-time hybrid mass spectrometer (QTOF), HPLC/diode-array detector (DAD), HPLC-UV, UHPLC-ESI-QqTOF-MS/MS	Luana et al. (2020)

Adulteration in Oils

There were various studies on the issues of adulteration in edible oils using Raman spectroscopy. Most of them use a combination of various techniques such as chemometric methods and other analytical methods apart from the application of Raman spectroscopy alone. The studies on the adulteration in oils in this review paper are focused on the adulteration of pure edible oils with other edible oils, the adulteration of foreign substances in edible oils, the adulteration of food products with oils and adulteration with waste cooking oil.

Adulteration of Pure Edible Oils with Other Edible Oils

Table 3 represents some studies on the adulteration of pure edible oils with other edible oils. Georgouli et al., 2017 conducted a study on the adulteration of EVOO with hazelnut oil by using Raman and Fourier transform infrared (FTIR) spectroscopy. The study found that continuous locality preserving projections (CLPP) and k-nearest neighbours (kNN) had obtained the best classification of the adulteration of EVOO at low percentages of hazelnut oil based on the chemical fingerprints of Raman and FTIR spectroscopy.

Best models of classification of pure EVOO and adulterated EVOOs were also obtained from the application of super vector machine – classification (SVM-C) and PLS from Raman data although four adulterated EVOOs were misclassified (Jiménez-Carvelo et al., 2017). Furthermore, Raman peaks at 1265 cm-1, 1441 cm-1 and 1657 cm-1 were used as the ratios of maximum Raman intensities to differentiate different types of vegetable oils and to identify the adulteration in EVOO (Farley et al., 2016). Detection of adulterated EVOO with cheaper oils such as canola, corn, grapeseed, sunflower and vegetable oils were identified at the lowest concentration of 5% adulteration in the study. Low limit of detection (LOD) and limit of quantification (LOQ) at 0.354% and 1.184% in monitoring the adulteration of argan oil with different concentrations of olive oil by using Raman spectroscopy combined with hybrid linear analysis developed by Goicoechea and Olivieri in 1999 (HLA/GO) had also been reported in a study (Joshi et al., 2019).

Raman spectroscopy obtained higher classification rates above 88% of sensitivity as compared to FTIR in classifying cold-pressed rapeseed oil adulteration with refined rapeseed oil and refined sunflower oil (McDowel et al., 2018). In a study by Mendes et al. (2015), good PLS prediction was achieved at the p-value of 0.117 by using Raman techniques to quantify the adulteration of soybean oil in olive oil. Philippidis et al. (2017) also found that the detection limit for characterization of Cretan EVOO adulteration with sunflower oil using Raman spectroscopy was lower than visible spectroscopy at 3.5%. Therefore, these studies have proven that Raman spectroscopy could be applied in the detection of adulteration in edible oils with other oils due to its high sensitivity and classification rates by combination of various chemometric methods.

There were similar studies on adulteration issues of edible oils using Raman spectroscopy such as a work by Tiryaki et al. (2016) which had accurately predicted the concentration of soybean oil adulteration in EVOO using PLS and determination of EVOO purity that had been adulterated with binary, ternary and quaternary mixtures using PLS by Duraipandian et al. (2019). Neves and Poppi (2017) also found that the adulteration levels above 2% in coconut oil were successfully quantified using Raman spectroscopy which had achieved satisfactory outcomes for the adulteration of coconut oil with sunflower, canola and Vaseline oils although the adulteration with babassu and palm kernel oils had higher error margin. Adulteration in EVOO with sunflower oil could also be distinguished by the intensity ratios of Raman spectral wavelengths at 1265 cm-1 to 1300 cm-1 and 1655 cm-1 to 1440 cm-1 since the ratio intensities increased as the adulteration level increased (Giubileo et al., 2015). Ryoo et al. (2017) reported that discrimination between pure and adulterated EVOOs was improved by maximizing the spectral differences with the measurement of temperature at -36.4 °C which led to a low error of discrimination at 8.0% using LDA and PCA.

There were higher changes in SERS intensity for detecting low concentrations of olive oil adulteration with bean oil were higher than conventional Raman intensity as the doping content increased (Du et al., 2019). Thus, this study would be useful to discriminate between different types of edible oil and adulteration.

Another technique using coherent anti-Stokes Raman spectroscopy (CARS) was conducted by Park et al. (2017) to determine the contents of adulterants such as perilla and corn oils or soybean oil in olive oil or sesame oil, respectively. The study was also included in determining the types of adulterants in blended olive oils using the intensity ratio as a comparative analysis. Apart from the adulteration of edible vegetable oil with other vegetable oils, there were also studies on the adulteration of other products such as the adulteration of clove essential oils (Jentzsch et al, 2017) and adulteration of animal fats or oils (Lee et al., 2018). Raman spectroscopy identified the types of adulterants in commercial clove essential oils such as benzyl alcohol and vegetable oil in three samples out of nineteen from the characteristics of the band at 1003 cm^{-1} for benzyl alcohol and from chemometric methods such as PCA and independent component analysis (ICA) for vegetable oil (Jentzsch et al, 2017).

Lee et al. (2018) applied Raman spectroscopy and intensity ratio techniques to classify four types of animal fats such as beef tallow, pork lard, chicken fat and duck oil and to detect the addition of pork lard in binary mixtures between the animal fats adulteration such as pork lard addition in beef tallow and duck oil. Hence, these studies are useful as they have applied some different techniques for detecting the types of adulteration in various types of oil and for the classification of adulterants in the products.

Table 3. Adulteration of pure edible oils with other edible oils, foreign substances in edible oils, food products with oils and waste cooking oil

Pure edible oils with other edible oils					
Product	Issue	Sub-Issue	Aim of the Study	Uses of Technique	References
EVOO	Adulteration	Addition of hazelnut oil	Identification of hazelnut oils in EVOO at low percentages based on spectroscopic chemical fingerprints	Raman spectroscopy and FTIR with CLPP and kNN	Georgouli et al. (2017)
EVOO	Adulteration	Addition of other edible oil	Differentiation of olive, soybean, corn, canola, sunflower, and cottonseed oil, to admixtures of these oils, and samples of aged soybean oil.	ESI-MS fingerprinting analysis	
EVOO	Adulteration	Addition of other vegetable oils	Discrimination of pure olive oil and adulterated olive oil	FTIR and Raman spectroscopy with kNN, PLS-DA, OCPLS, SVM-C, SIMCA and PLS-R	Jiménez-Carvelo et al. (2017)
EVOO	Adulteration	Addition of canola, corn, grapeseed, sunflower and vegetable oil	Detection of adulteration in EVOO with different concentration of other edible oils	Raman spectroscopy with peak intensity ratios	Farley et al. (2016)
Argan oil	Adulteration	Addition of olive oil	Determination of different adulteration levels of olive oil in argan oil	Raman spectroscopy with NAS-based HLA/GO	Joshi et al. (2019)
Canola oil	Adultertaion	Addition of vegetable oils such	Detection of vegetable oils such as sunflower,	GC-IMS and Chemometric analysis	Chen et al. (2018)

		as sunflower, soybean, and peanut oils	soybean, and peanut oils in canola oil		
Cold pressed rapeseed oil	Adulteration	Addition of refined rapeseed oil and refined sunflower oil	Detection of refined rapeseed oil and refined sunflower oil substitution fraud in cold pressed rapeseed oil	Raman spectroscopy with PCA, SIMCA, PLS-DA, LDA-KNN, LDA-SVM and PLS-R	McDowell et al. (2018)
EVOO	Adulteration	Addition of soybean oil	Quantification of adulteration of EVOO with soybean oil	NIR, MIR and Raman spectroscopy with PLS	Mendes et al. (2015)
EVOO	Adulteration	Addition of sunflower oil	Investigation of adulteration of Greek (Cretan) EVOO with sunflower oil based on the characterization of spectral properties	Raman spectroscopy with PLS	Philippidis et al. (2017)
EVOO	Adulteration	Addition of soybean oil	Prediction of the percentage level of soybean oil adulteration in olive oil	Raman spectroscopy with PLS	Tiryaki et al. (2016)
EVOO	Adulteration	Addition of corn oil, rapeseed oil and soybean oil	Quantification of the olive oil purity regardless of the number of adulterants	Raman spectroscopy with PLS	Duraipandian et al. (2019)
EVOO	Adulteration	Addition of DNA	Detection of adulterated olive oil	DNA-based approaches	Kalaitzis et al. (2016)
Coconut oil	Adulteration	Addition of sunflower oil, Vaseline oil, babassu oil, canola oil and palm kernel oil	Evaluation of the adulteration and purity in coconut oil	Raman spectroscopy with MCR-ALS	Neves et al. (2017)

EVOO	Adulteration	Addition of sunflower oil	Quantification of adulteration of EVOO with sunflower oil	FTIR and Raman spectroscopy with intensity ratio	Giubileo et al. (2015)
EVOO	Adulteration	Addition of soybean oil	Differentiation between EVOO and adulterant (soybean oil)	Raman spectroscopy with PCA and LDA	Ryoo et al. (2017)
Edible vegetable oil products	Adulteration	Addition of other vegetable oil and oil oxidation analysis	Discrimination of edible oil type, oxidation and adulteration	SERS with PCA	Du et al. (2019)
Edible oils	Adulteration	Addition perilla oil or canola oil into EVOO and addition of soybean oil into sesame oil	Determination of adulteration in edible oils	CARS with intensity ratio	Park et al. (2017)
Clove essential oil	Adulteration	Addition of benzyl alcohol and vegetable oil	Identification of adulteration in clove essential oil and the assessment of its quality	Raman spectroscopy with PCA and ICA	Jentzsch et al. (2017)
Beef tallow and duck oil	Adulteration	Addition of pork lard	Detection of pork lard in beef tallow and duck oil	Raman spectroscopy with simple calculation of intensity ratios	Lee et al. (2018)

Foreign substances in edible oils					
Product	Issue	Sub-Issue	Aim of the Study	Uses of Technique	References
Soybean oil, olive oil, sunflower oil and commercial vegetable oils	Adulteration	Addition of copper chlorophyll	Detection of copper chlorophyll in vegetable oils	Surface-enhanced Raman spectroscopy (SERS)	Lian et al. (2015)
Essential oils from basil, cinnamon leaves, citronella	Adulteration	Addition of butylated hydroxyanisole (BHA)	Determination of BHA in various types of edible oils and essential oils	SERS	Wrona et al. (2015)

grass, cumin seeds, dill seeds, ginger, clove, oregano, rosemary and thyme, commercial sunflower oil and olive oil					
Soybean oil	Adulteration	Addition of benzo(a)pyrene (BaP)	Evaluation of BaP concentration in edible oil	SERS	Fu et al. (2015)
Canola oil	Adulteration	Addition of erucic acid	Determination of erucic acid percentage in the total fatty acids in canola oil	Raman spectroscopy and GC with PLS regression	Velioglu et al. (2017)
Corn oil, gutter oil	Adulteration	Addition of capsaicin	Detection of capsaicin in corn oil and determination of gutter oil	SERS with DFT	Tian et al. (2017)
Edible oils	Adulteration	Addition of trielaidin	Quantification of different trielaidin concentrations in edible oils	Raman spectroscopy with SMLR and PLS	Gong et al. (2019)
Olive oil and sunflower seed oil	Adulteration	Concentration of triglycerides of oleic and linoleic acids	Assessment of adulteration of olive oil with sunflower seed oil by changing the ratio of oleic and linoleic acids	Raman spectroscopy with relative intensity ratio	Berezin et al. (2018)
Corn oil, oil-in-water emulsion	Adulteration	Addition of α-tocopherol (TOC)	Characterization of bioactive component, TOC loaded in oil-in-water emulsion	Raman microspectroscopy and HPLC with polynomial fitting algorithm	Wang et al. (2018)
Vegetable oil	Adulteration	Adding a minuscule amount of synthetic DNA	Identification of adulterated vegetable oil	PCR and DSC	Abbas et al. (2016)

Food products with oils					
Product	Issue	Sub-Issue	Aim of the Study	Uses of Technique	References
Dairy cream and cream-like products	Adulteration	Addition of vegetable oils such as sunflower oil, coconut oil and palm oil	Discrimination between dairy cream and cream-like products prepared with vegetable oils	Raman spectroscopy with PCA-DA	Nedeljkovic et al. (2017)
Milk cream and yogurt	Adulteration	Addition of vegetable fats or oil such as corn oil, sunflower oil, margarine	Discrimination of non-milk-based fats or oil in milk cream and yogurt as a dairy product	Raman spectroscopy with PCA	Karacaglar et al. (2019)
Ghee (milk fat, cream or butter)	Adulteration	Addition of palm oil and beef tallow	Interaction of different geographical regions and adulterant levels on chemical profile and spectral detection of ghee	Gas chromatography and Raman spectroscopy with RSM	Erfani et al. (2019)
Waste cooking oil					
Product	Issue	Sub-Issue	Aim of the Study	Uses of Technique	References
EVOO	Adulteration	Addition of waste cooking oil	Detection of waste cooking oil in EVOO	Raman spectroscopy with iPLS and SiPLS	Li et al. (2017)
Palm oil	Adulteration	Detection of recycled cooking oil	Detection of fresh palm oil with recycled cooking oil	FTIR Spectral analysis	Lim et al. (2018)

Adulteration of Foreign Substances in Edible Oils

Detection of foreign substances in edible oils could also be one of the adulteration issues. Lian et al. (2015) reported on the detection of copper chlorophyll in vegetable oils by validating the peaks of copper pyropheophytin α at 752 cm^{-1} and 990 cm^{-1} in vegetable oils with a detection limit of 5 ppm. It was successful in detecting the presence of copper chlorophyll in commercial vegetable oils by a ratio of 21/23. The adulterated vegetable oils with copper chlorophyll were a commercial fraud as they could be disguised as expensive vegetable oils. Meanwhile, the addition of butylated hydroxyanisole (BHA) was applied to prevent food fats and oils oxidation. Wrona et al. (2015) successfully determined the BHA contents in sunflower, basil and oregano oils using SERS by studying the characteristics of BHA peaks and the effects of temperature measurement. Benzo(a)pyrene (BaP) is a toxic substance that could be produced from incomplete combustion (Fu et al., 2015). The SERS could detect the different concentrations of trace BaP in soybean oil as well as in real oil samples. Furthermore, erucic acid in the total fatty acids of canola, mustard seed and rapeseed oils could risk human health (Velioglu et al., 2017).

Velioglu et al. (2017) found the LOD and LOQ values at 1.76% and 8.81% for the detection of erucic acid contents in the pure and adulterated canola and mustard seed oils. In a study by Tian et al. (2017), the detection of capsaicin in cooking oil such as corn oil was identified to distinguish between cooking oils and gutter oils. The study produces prominent peaks characteristic of capsaicin at 807 cm^{-1} and 1264 cm^{-1} with an LOD of 1 mg/L. There was a study on the detection of trans unsaturated fatty acids such as trielaidin in edible oils that could risk human health (Gong et al., 2019). The study could differentiate between cis fatty acid at 1654 cm^{-1} and trans fatty acid at 1667 cm^{-1} and identify the increase in concentrations of trielaidin in edible oils based on the decrease of Raman intensity at 1653 cm^{-1}. Thus, these studies show that there were a lot of different techniques for detecting foreign substances in edible oils based on their characteristics and properties.

Moreover, Raman bands at 1660 cm^{-1} and 1445 cm^{-1} were studied by using relative intensity ratios to determine the concentration of triglycerides of oleic and linoleic acids such as α-linolenic, arachidonic, EPA and DHA in cold-pressed olive and sunflower seed oils (Berezin et al., 2018). The study evaluated the adulteration of olive oils with sunflower oils based on the changes in ratios of oleic and linoleic acid contents. Wang et al. (2018) also conducted a study that identified α-tocopherol (TOC) contents in corn oil and emulsion with Raman intensity at 481.9 cm^{-1} and 588.6 cm^{-1}. The study showed that the LODs were 5.1 and 21.2 g/kg to the respective Raman intensity of corn oil and the standard deviation values were 4%–16% and 2%–6% to the respective Raman intensity of the emulsion. Therefore, the studies focused on the determination of the various types of component contents in edible oils to evaluate the concentration of adulteration in the edible oils. **Table 3** has summed up some studies on adulteration of foreign substances in edible oils using Raman spectroscopy.

Adulteration of food products with oils

There were also studies on the adulteration of food products with oils (**Table 3**). There were many fraud cases such as the addition of vegetable oils to dairy products to increase profits and economic gain since vegetable oils are cheaper than milk fats. Due to this case, Karacaglar et al. (2019) conducted a study using Raman spectroscopy to determine and distinguish between the cheaper fats or oils such as corn oil, margarine, sunflower oil and vegetable fat blends in milk cream and yogurt. The study suggested that the discrimination between the fats and cheaper oils was successfully identified based on the differentiation of the Raman spectra with the aid of chemometric methods such as PCA. A similar study was also previously conducted by Nedeljkovic et al. (2017) that distinguished dairy cream products from cream-like substitutes with vegetable oils such as sunflower, coconut and palm oils. A high sensitivity model at 100% using PCA-DA

detection for adulteration of dairy cream with vegetable fats or oils. Another study was conducted on ghee which is a mixture of milk fat, cream or butter (Erfani et al., 2019). The study reported that the adulteration of ghee with palm oil had produced a detection level of higher than 18.25% based on the Raman bands at 1298 to 1305 cm^{-1} and 1426 to 1460 cm^{-1}. Hence, these studies have proved that Raman spectroscopy is capable of identifying different types of adulterations, especially in distinguishing various types of edible oils including the presence of foreign oils in other food products.

Adulteration with Waste Cooking Oil

In studies on the adulteration of edible oils with waste cooking oil using Raman spectroscopy conducted by Li et al. (2017), the different concentrations of waste cooking oil in the EVOO resulted in high prediction accuracy of the model. Both lower limit of application (LLA) and LOD were obtained at 0.5% and 0.475% based on linear regression. This showed that Raman spectroscopy was capable of distinguishing pure EVOO and adulterated EVOO with waste cooking oil. The study of adulteration of edible oil with waste cooking oil has been tabulated in **Table 3**.

Conclusion

Due to various cases of adulteration, Raman spectroscopy has been applied to identify the adulteration in food products especially edible oils to determine the authenticity of pure edible oils in the market. Authentication of oils was discussed based on the qualitative and quantitative analysis of edible oils such as fatty acid profile, harvest year, variety and geographical origin of the edible oils. The content of bioactive components in the edible oils was also studied to authenticate different types of oil. Raman spectroscopy is also capable of detecting and distinguishing different types of adulteration cases of edible oils.

A lot of previous studies were focused on the application of Raman spectroscopy in detecting the adulteration of edible oil with other edible oils, adulteration of foreign substances in edible oils, adulteration of food products with vegetable oils and adulteration of edible oil with waste cooking oil. However, the success of these studies was mainly aided by various other techniques such as chemometric methods and other comparative analysis methods. Thus, advancement in the methods that could provide the most accurate prediction of classification between edible oils is significant to aid with the application of Raman spectroscopy in future studies

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