REVIEW

Edible Oils Adulteration: A Review on Regulatory Compliance and Its Detection Technologies

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Abstract: Various events of edible oils adulteration with inferior ingredients were reported regularly in recent years. This review is aimed to provide an overview of edible oils adulteration practices, regulatory compliance and detection technologies. Many detection technologies for edible oils adulteration were developed in the past such as methods that are based on chromatography or spectroscopy. Electrochemical sensors like electric nose and tongue are also gaining popularity in the detection of adulterated virgin olive oil and virgin coconut oil. It can be concluded that these detection technologies are essential in the combat with food adulterers and can be improved.

Key words: edible oil adulteration, regulatory compliance, chromatography, spectroscopy, electronic tongue and nose

1 Introduction

Edible oils are defined as a food commodity of any kind of origin, source or structure that is manufactured for human intake entirely or in portion from a fat or oil other than dairy products¹⁾. To prepare delicious food, edible oils are often used due to its texture and exceptional flavour attributes. For many years, people utilized oil to make the flavour of the food better²⁾. It is one of the indispensable nutrition components in our daily diet³⁾. The overall production of edible oil in India was 25.3 million tonnes in 2015-16 and the total area under edible oils was 26.13 million hectares. Total production of edible oils was recorded in 2013-14, which amounted to 32.75 million tonnes from 28.05 million hectares. India imported 148.2 metric tonnes of edible oils in 2015-16 and net domestic availability was 86.37 metric tonnes⁴⁾.

Adulteration in food commodities is a frequently faced problem in the current food business. Nearly all food commodities are subjected to quality problems, especially those having high commercial values⁵⁾. The substitution of expensive materials with cheaper ingredients can be very

profitable for a producer or trader of raw ingredients since vending the product as if it remained pure permits profit values to be highly amplified⁶⁾. Owing to the growing demand of edible oils on the domestic and foreign markets, high-priced oil adulterated with low-priced oil is a major issue that can further threaten consumer health. For instance, extra virgin olive oil (EVOO), which is the highest quality of olive oil, is the most prone to fraudulent activity⁷⁾. In addition to possibly preventing olive oils from meeting extra virgin requirements, adulteration will reduce the presence of EVOO in a product and can in turn reduce its associated health benefits and flavour qualities⁷⁾.

Unscrupulous businessmen also adulterate high-quality oil with used frying oil, which is a potential health risk for consumers⁸⁾. Consumers are facing all kind of health issues while consuming adulterated foods because they cannot differentiate between real and adulterated foods with their naked eyes⁸⁾. Therefore, food-related organisations need to establish and use effective methods to detect such adulterations, which will then ensure the authenticity and purity of edible oils and fats. Advanced and suitable techniques

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Accepted June 2, 2021 (received for review March 27, 2021)

Journal of Oleo Science ISSN 1345-8957 print / ISSN 1347-3352 online

<http://www.jstage.jst.go.jp/browse/jos/> <http://mc.manuscriptcentral.com/jjocs>

are being developed and currently being used to identify adulteration in edible oils that can help reduce consumer health risks. This paper provides a comprehensive review on the adulteration of edible oils and their detection methods. The regulatory compliance of edible oils in different countries will also be outlined.

2 Regulatory Compliance of Edible Oils

Food regulations include the legislative authority and the necessary legal structure for food control activities. The main objective is to protect the health of consumers from fraud by ensuring the important quality and health of food. According to Malaysian Food Regulation 1985, it stated that edible oils and fats must not contain adulterants, rancidity, offensive odour and taste. Edible oil in its single form shall be free from admixtures with other fats or oils. However, permitted food conditioners and antioxidants are allowed to be applied to the edible oils and fats as stated in the regulations. Blending of edible oils is acceptable as long as it meets the standard specifications established for edible oil in the regulations and cannot contain more than 30 g/kg of unsaponifiable content. There are different standards for different kinds of edible oils stated in Malaysia Food Regulation 1985, as shown in Table 1.

In Thailand, edible fats and oils must be free from foreign matter and rancid odour in compliance with the requirements stated out in the regulations⁹. The blending of edible fats and oils consisting of two or more different types of fats and oils is permitted as long as the processes are approved by the Thailand Food and Drug Administration (Thai FDA) either by mechanical process, inter-esteri-

fication process or by other processes. Moreover, the action of blending edible oils shall comply with the additional standard or specification approved by the Thai FDA, as the condition may be⁹. As far as India is concerned, the blending of edible oils is permitted as long as the proportion by weight of any edible vegetable oil used in the blend is not less than 20%¹⁰. It is allowed as long as the edible oils comply with the respective requirements established in the regulations. The blend must be clear and also be free from rancidity, suspended or insoluble matter or any other foreign matter, separated water, added colouring matter, flavouring substances, mineral oil, or any other animal and non-edible oils, or fats, argemone oils, hydrocyanic acid, castor oil and tricresyl phosphate¹⁰.

In Brunei, the modification of fats and oils in edible oils and fats is permitted, and these foods are usually recognised as healthy¹¹. Unless otherwise stated, edible fats and oils must have a peroxide value of no more than 10 milliequivalents of peroxide oxygen per kilogram of fat or oil¹¹. They may also contain permitted antioxidants. There are no regulations stating whether or not the blending of edible oils is permitted or prohibited. There are, however, requirements for vanaspati, a desi vegetable ghee made from a mixture of groundnut oil, cotton seed oil, sesame oil, or a combination of these oils, as well as other vegetable oils that are safe to eat¹¹. As a result, blending of harmless vegetable oil is permitted to make vanaspati as long as it complies with the respective requirements established in Brunei's Public Health (Food) Regulations.

According to Codex Alimentarius Commission¹², food additives such as colouring in edible oils and fats are permitted to restore the natural colour lost during processing or for colour standardisation purposes without misleading the user. Flavoring is also permissible as long as the ingre-

Table 1 Standards for different edible oils stated in Malaysia Food Regulation 1985.

Edible oil	Specific gravity	Refractive index	Saponification value (mg)	Iodine value	Unsaponifiable matter (g/kg)
Coconut oil	0.915-0.920	1.448-1.449	248-264	7.5-10.5	<5
Corn oil	0.917-0.925	1.465-1.480	187-195	103-130	<28
Cottonseed oil	0.915-0.928	1.458-1.474	189-198	99-119	<15
Peanut oil	0.914-0.917	1.460-1.465	187-196	80-106	<10
RBD palm oil	0.8900-0.8932	1.450-1.460	190-209	50-55	<10
Olive oil	0.910-0.916	1.4677-1.4705	184-196	75-94	<15
Rice bran oil	0.910-0.920	1.4600-1.4700	175-195	90-105	<30
Rapeseed oil	0.910-0.920	1.465-1.469	168-181	94-120	<20
Safflower seed oil	0.922-0.927	1.467-1.470	186-198	135-150	<15
Sesame seed oil	0.915-0.923	1.465-1.469	187-195	104-120	<20
Sunflower seed oil	0.918-0.923	1.467-1.469	188-194	110-143	<15
Soya bean oil	0.919-0.925	1.466-1.470	189-195	120-143	<15

dients used comply with the requirements specified in the Codex Alimentarius Committee¹²⁾. However, it is mentioned in Codex Alimentarius Commission that no additives can be applied to virgin or cold pressed oils so as not to alter the oil¹²⁾. Virgin and cold pressed oils can only be obtained by mechanical procedures only, expelling or pressing, without the application of heat. After that, they may also have been purified by washing with water, settling, filtering and centrifuging only. There is no mention, however, of whether or not the mixing of edible oils and fats is permitted. Table 2 shows the standards for different edible oils stated in Codex Alimentarius Commission¹²⁾.

3 Types of Edible Oils Adulteration

Adulteration of edible oil is not a new issue and can happen in any given area or country. One of the reasons for edible oil adulteration is to capitalise on consumer demands and raise profits. Products with lower economic value are often mixed with higher priced commodities to improve on profits. For example, from an economic point of view, replacing high priced oil with cheaper oil is so profitable for producers and encourages them to do so¹³⁾. One of the most reported cases was the mixing of cottonseed oil imported into Egypt with palm oil produced in Malaysia¹⁴⁾. Another case based on Rossell, was the reconstitution of palm oil by mixing linpalm stearin and palm oil in a Singapore cocktail¹⁴⁾. By replacement of more expensive

oils and fats with cheaper one, often result in a blend deviating from the mixture proportions displayed on the label, or if the blend is traded as authentic, the vendor will ultimately commit fraud¹⁵⁾. This will also have an effect on honest suppliers who suffer unfair competition¹⁵⁾.

On the other hand, minimally processed foods have gained broad interest in most of the countries. Since many people claim that these foods have superior nutritional qualities, mainly because their “naturalness” can be achieved to a higher degree by intentionally avoiding conventional food processing techniques such as refining¹⁵⁾. Cold press oil obtained by mechanical means is usually manufactured using only the highest quality raw materials, otherwise it can cause deterioration during storage¹⁵⁾. Since there is no contact between cold press oil and chemicals and even solvents during refining, the market for cold press vegetable oils is therefore growing¹⁶⁾. Cold press oil is more costly than refined oil, there is a tendency to combine it with refined oil to lower production costs in order to gain extraordinary profits.

There are also loose edible oil adulterations across the country. One of India’s leading consumer organisations, Consumer Voice, conducted a study that reported up to 85% adulteration in loose edible oil sold in India across 15 states¹⁷⁾. Eight major varieties of edible oil, namely mustard, sesame, coconut, sunflower, palm olein, soybean, groundnut and cottonseed oil, were found to have been adulterated in this study. Coconut oil samples were found to be adulterated at 85% while cottonseed oil, sesame oil

Table 2 Standards for different edible oils stated in Codex Alimentarius Commission¹²⁾.

Edible oil	Relative density	Refractive index	Saponification value (mg)	Iodine value	Unsaponifiable matter (g/kg)
Coconut oil	0.908-0.921	1.448-1.450	248-265	6.3-10.6	<15
Arachis oil	0.912-0.920	1.460-1.465	187-196	86-107	<10
Maize oil	0.917-0.925	1.465-1.468	187-195	103-135	<28
Olive oil	0.910-0.916	1.4677-1.4705	184-196	75-94	<15
Babassu oil	0.914-0.917	1.448-1.451	245-256	10-18	<12
Palm oil	0.891-0.899	1.454-1.456	190-209	50-55	<12
Palm kernel oil	0.899-0.914	1.448-1.452	230-254	14.1-21.0	<10
Palm olein	0.899-0.920	1.458-1.460	194-202	<56	<13
Palm stearin	0.881-0.891	1.447-1.452	193-205	<48	<9
Cottonseed oil	0.918-0.926	1.458-1.466	189-198	100-123	<15
Grapeseed oil	0.920-0.926	1.467-1.477	188-194	128-150	<20
Rapeseed oil	0.910-0.920	1.465-1.469	168-181	94-120	<20
Safflower seed oil	0.922-0.927	1.467-1.470	186-198	136-148	<15
Sesame seed oil	0.915-0.924	1.465-1.469	186-195	104-120	<20
Sunflower seed oil	0.918-0.923	1.461-1.468	188-194	118-141	<15
Soya bean oil	0.919-0.925	1.466-1.470	189-195	124-139	<15

and mustard oil were not any better with 74.07%, 74% and 71.77% adulteration, respectively, found in this study¹⁷⁾. In many cases, it has been found that mineral oil, karanja oil, castor oil, and artificial colours are heavily used in edible oil adulterations¹⁸⁾.

4 Edible Oil Adulteration and Its Effects on Health

Edible oil often becomes impure and unfit for human consumption as a result of adulteration. It has been reported that adulteration of edible oils resulted in severe health issues in some cases. Azadmard-Damirchi and Torbati reported a Spanish toxic oil syndrome or Spanish olive oil disease, which led to over 600 deaths because non-edible rapeseed oil was sold as an edible oil and even as olive oil¹⁶⁾. Further health problems may also arise if an adulterant used belongs to a specific type of allergens such as peanut oil, as certain buyers may be sensitive to the peanut proteins in the adulterant⁷⁾.

Adulteration of argemone mexicana seed oil in edible oils has been identified in a number of cases as a cause of epidemic dropsy since it contains Sanguinarine and Dihydrosanguinarine, toxic agents (alkaloids) found in Argemon¹⁾. In addition, argemone oil mix with edible oils will not only cause epidemic drowsiness but also glaucoma and loss of eyesight¹⁹⁾. It is also reported that edible oil adulterants, argemone oil and butter yellow can cause cancer of the gallbladder²⁰⁾.

Loose edible oil adulterations run the risk of cancer, paralysis, liver damage and cardiac arrest as such oils are heavily adulterated. In the study of Navya *et al.*, it is said that edible oils adulterated with castor oil cause stomach problems¹⁸⁾. On the other hand, edible oils that are adulterated with mineral oils can cause liver damage and cause carcinogens¹⁸⁾. As a result, with a few recorded cases of health hazards associated with edible oil adulteration, food safety is critical, as with fats and oil authenticity issues.

5 Technologies for Authentication of Edible Fats and Oils

5.1 Chromatography method

The authenticity of edible oils is commonly evaluated using analytical chemistry methods for the determination of fat phase components. Analysis of the fatty acid composition by gas chromatography is the most widely used method (GC)²¹⁾. Based on study from Andrikopoulos *et al.*, olive oil typically contains zero or has very low levels (< 0.5%) of these two triglyceride compounds, trilinolein and tripalmitin²²⁾. Detection of rising amounts of trilinolein or tripalmitin in olive oil by capillary gas chromatography (CGC) with a flame ionisation detector (FID) was used in

this study to identify adulteration of olive oil with several seed oils (corn, cottonseed, palm, soybean, and sunflower).

According to Uncu *et al.*, in order to authenticate the botanical origin of olive oil, the chromatographic technique is used to determine whether the fatty acid composition of 11 plant species of edible oil samples deviated from that of olive oil, as mentioned in the Codex Alimentarius Standard²³⁾. Linoleic acid was indicated as a fraud determinant to detect adulteration for all mixing ratios evaluated with values that exceeded the threshold of the 1% as described in the Codex Alimentarius Standard for olive oil. In Jafari *et al.*, fatty acid profiles of olive oil adulterated with three cheap oils, soybean, sunflower, and canola oils were obtained by performing gas chromatographic analysis of fatty acid methyl ester (FAME)²⁴⁾. Analyzing the fatty acid profiles obtained through gas chromatographic analysis of FAME, the adulterated olive oils reported higher levels of linolenic and linoleic acids, but significantly lower levels of oleic acids relative to genuine olive oils²⁴⁾. Jabeur *et al.* have also demonstrated that using gas chromatography and high-performance liquid chromatography to obtain the fatty acid composition of EVOO and adulterated oil samples (Fig. 1)²⁵⁾. It has been suggested that the linolenic acid content observed in the fatty acid compositions could be used as a parameter for the detection of EVOO adulterated with different levels of soybean oil. It has also been shown that the adulteration can be identified by the increase of the *trans*-fatty acid contents with 3% of soybean oil, 2% of corn oil, and 4% of sunflower oil²⁵⁾.

The capability of solid-phase microextraction (SPME) for the determination of volatile constituents in vegetable oils has occasionally been recognized in the literature by various authors who have insisted on the need to carefully optimise the experimental variables involved in the distillation process²⁶⁾. Krist *et al.* identified the adulteration of poppy seed oil with sunflower oil using the solid phase microextraction gas chromatography/mass spectrometry (SPME-GC-MS) method²⁷⁾. The analytical method SPME was established in the 1990s as a means of extracting and pre-concentrating pollutants in water²⁸⁾. By using R-pinene as a marker compound, this method, together with the analysis of FAMES by GC-MS, was able to detect the mixing of sunflower oils in all relevant quantities (5–40%) in poppy seed oil. Solid phase microextraction and multidimensional gas chromatography were also used in detecting adulteration of olive oil of different varieties with virgin hazelnut oils in percentages of up to 7%²⁹⁾. This method can be used to detect the presence of R- and S-enantiomers of filbertone as an indicator in olive oils adulterated with refined hazelnut oil²⁹⁾.

Figure 2 shows the full GC-sterol profile of argan oil. Sterols are useful candidates for identification of edible oil adulteration³⁰⁾. Campesterol is a sterol present in seed oils, which contributes less than 0.4% of the overall sterol

Fig. 1 Gas chromatogram showing the FAMEs profiles of Chemlali EVOO (a) and binary mixtures containing 90% EVOO and 10% of either soybean (b), sunflower (c), or corn (d) oils using GC-FID²⁵⁾. *cis*- and *trans*-FAMEs were identified a comparison of their retention times versus pure standards analyzed under the same conditions. They were quantified according to their percentage area, obtained by the integration of the peaks. The results were expressed as the percentages of individual fatty acids in the lipid fraction. The linolenic acid percentage could be used as a parameter for the detection of EVOO fraud as well as by the increase of *trans*-fatty acid contents in the adulterated EVOO.

content of pure argan oil³¹⁾. Hence, campesterol is selected as the adulteration marker in this research. The method, GLC FID-capillary column in compliance with NP EN ISO 12228:1999 was used to assess the amount of campesterol at a concentration ratio of 99/1, 98/2, and 95/5 for mixtures of argan oil and common oils. Evidently, for blending of campesterol-rich common oils with argan oil, campesterol levels above 0.4% were observed at an adulteration level of 1% as predicted from the campesterol values measured. This method also assists low campesterol-containing oils in establishing a 95% purity label for olive oil and hazelnut oil as well as a 98% purity label for apricot oil. In the research of Salghi *et al.*, high-performance liquid chromatography is performed to obtain triacylglycerol profiles as an indication of adulteration of argan oils with up to 5% of sunflower,

soya bean and olive oil³²⁾. This study shows that using high-performance liquid chromatography in conjunction with evaporative light scattering allows researchers to conduct rapid sampling for authenticity assessment. Chromatography methods, however, have number of disadvantages, the most important of which is that they often require complex sample pretreatments which is time consuming. Besides that, these methods require highly qualified personnel, and they are insufficient for screening such adulterated oils with identical fatty acid compositions³³⁾.

5.2 Spectroscopy method

Spectroscopic methods can also be used to detect adulteration in edible oils, as these methods have the advantage that the time-consuming preparation of the sample is

Fig. 2 Full GC-sterol profile of argan oil³¹⁾. The x-axis of the gas chromatogram shows the amount of time taken for the analytes to pass through the column and reach the mass spectrometer detector. Campesterol retention time (RT = 24.17 min) was clearly distinct from that of other sterols in the GC sterol chromatogram (stigma-8,22-dien-3-ol: RT = 26.42 min, spinasterol: RT = 28.16 min, schottenol: RT = 31.10 min, stigmasta-7,24-dien-3-ol: RT = 31.94 min). Sterol quantification was achieved by use of an internal 0.2% chloroform solution of α -cholestanol. In these conditions, campesterol can be identified through a comparison of a retention time of 24.19 min. Determination of the campesterol level, a sterol found in seed oils, represents less than 0.4% of argan oil total sterol content can be used as the major analysis method to assess unambiguously argan oil purity up to 98%. The results showed that the argan oil sample in this study contained 0.29% of campesterol.

usually kept to a minimum. Fourier Infrared Transform (FTIR) spectroscopy has been commonly used in food-related science for more than 15 years. Based on study from Ozulku *et al.*, ATR-FTIR spectroscopy combined with chemometric, principal component analysis (PCA) and cluster analysis allowed detection of adulteration of cold-pressed sesame oil with hazelnut, canola, and sunflower oils in different concentrations ranged from 1 to 50%³⁴⁾. Rohman and Che Man has also stated that FTIR spectroscopy can be used as a non-destructive and fast technique for the determination of adulteration of cod-liver oil with different concentrations of lard (0.5–50% v/v in cod-liver oil)³⁵⁾. FTIR combined with a multivariate analysis was also used to detect and measure the adulteration of avocado oils with various edible oils such as sunflower, soybean and canola³⁶⁾. Soft Independent Modeling Class Analogy (SIMCA) model was used to discriminate between avocado oil and adulterants (sunflower, canola and soybean oils) based on their chemical analysis (peroxide and iodine value) showed 100% correct classification rate of the edible oil samples³⁶⁾.

Raman spectroscopy was combined with chemometric methods PCA, principal component regression (PCR), partial least squares (PLS) and artificial neural networks (ANNs) to detect, classify and quantify the adulteration of butter with margarine (Fig. 3)³⁷⁾. On the other hand, EVOO samples adulterated with soybean oil, corn oil and sunflower seed oil can also be characterized by using Raman spectroscopy (1000–1800 cm^{-1}) and chemometrics, by employing external standard method (ESM) for the quantitative analysis, which was compared with the results obtained by support vector machine (SVM) methods³⁸⁾. Fourier transform (FT)-Raman spectroscopy combined with multivariate procedures has been described as a tool for olive oil au-

thentication³⁹⁾. PCR showed 100% correct discrimination between authentic and adulterated samples and 91.3% correct classification at various adulteration levels³⁹⁾. Raman spectroscopy with excitation in the visible spectral range coupled with partial least-squares (PLS) regression is a rapid and nondestructive method for monitoring adulteration of extra-virgin olive oil with different levels of sunflower oil ranging from 5 and 100%⁴⁰⁾.

On the other hand, as it has a low signal-to-noise ratio with rapid spectral acquisition, fluorescence spectroscopy is said to have an advantage over Raman and other spectroscopic techniques. In addition, its sensitivity is also improved by excellent selectivity, which further contributes to its advantages. According to Ali *et al.*, fluorescence spectroscopy (excitation wavelength at 350 nm) was applied to identify the adulterant with sunflower oil in pure EVOO in combination with PCA and PLS regression⁴¹⁾. In this study, the EVOO is compared and distinguished from sunflower oil by obtaining a fluorescent spectrum containing chlorophyll and carotenoid fingerprints which are characteristics of EVOO⁴¹⁾.

Next, synchronous fluorescence spectroscopy (250–800 nm) combined with three different multivariate methods, namely PCA, soft independent modeling of class analogies (SIMCA) and PLS regression were also used in the study, Karuk Elmas *et al.* to detect adulteration of cold-pressed grapeseed oil with refined soya bean oil at different adulterant levels from 5 to 50%⁴²⁾. Based on the study, Ntakatsane *et al.*, fluorescence spectroscopy and gas chromatography coupled with PCA and 3-way PLS regression were used in detecting adulteration of pure butterfat with different vegetable oils at various concentrations ranging from 5%–40%⁴³⁾. The detection is achieved by distinguishing

Fig. 3 Original (a) and first-derivative of the Raman spectra (b) of the samples. A: Margarine, B: Butter (20%) + Margarine (80%), C: Butter (80%) + Margarine (20%), D: Butter³⁷. The baseline spectra of adulterated samples and the intensity of band ranges between 380 and 565 cm^{-1} increased together with the margarine concentrations. In the case of margarine, ratios in adulterated samples were higher, and the spectrum of bands ranging between 570 and 760 cm^{-1} began to change visibly and two bands (608 and 730 cm^{-1}) belonging to butter disappeared. Raman spectra of margarine change due to the source of edible oils and added ingredients that cause fluorescence.

between the pure and adulterated samples on the basis of the total concentration of saturated fatty acids and unsaturated fatty acids in the fatty acid compositions, as well as the three main fluorophores: tryptophan, tocopherols and riboflavin.

Chen *et al.* reported that Near-infrared (NIR) technique combined with competitive adaptive reweighted sampling (CARS) or elastic component regression (ECR) is proved to be feasible for rapidly detecting sesame oil adulterated with other vegetable oils (Fig. 4)⁴⁴. Basri *et al.* utilized the short-wave NIR method in two different scan modes, transreflectance and transmission mode to compare the accuracy for detecting the presence of lard adulteration in palm oil⁴⁵. Based on the result of classification and quantification analysis using soft independent modelling class analogy (SIMCA) and PLS regression, the transmission mode has yield better prediction model compared to the transreflectance mode to classify the pure and adulterated palm oil⁴⁵.

Three experimental setups with PLS regressions, based on near infrared technology (NIR), included a commercial portable NIR, coupled to both reflexion and immersion probes, and a prototype of a multichannel Quasi Imaging Visible NIR spectrometer (QIVN) coupled to an immersion probe were tested for rapid “at-line” assessment of sunflower oil adulterated with different percentages of mineral oil range from 0.5% to 10% in work of Picouet *et al.*⁴⁶. Commercial portable NIR, coupled to both reflexion and immersion probes has been shown as the best among the other two experimental setups since it provides satisfactory calibrations and low number of false positives starting

Fig. 4 Near-infrared spectra of sesame oil adulterated by other oils⁴⁴. By a visual inspection, useful absorption are located in the region of 5600–5900 cm^{-1} . The two peaks around 5791 cm^{-1} and 5677 cm^{-1} mainly rise from the first and second overtones of CH stretching vibrations of CH, CH₂, CH₃, and CH = CH in glycerides. The compositions of the vegetable oils vary with the different fatty acids in the glyceride molecules. Different oils have different fatty acid composition and there should be differences in the intensities of bands arising from the overtones and combinations of the CH₃ and CH₂ stretching vibrations.

from levels of mineral oil around 1%⁴⁶⁾.

Furthermore, Irfan *et al.* proposed an interesting handheld NIR spectrometer to detect adulteration palm cooking oil with recycled cooking oil (RCO)⁴⁷⁾. The NIR spectrum calibration was performed using PCA to classify the level of adulteration, i.e. qualitative approach. The result showed that the classification accuracy was 100% when the concentrations of the recycled cooking oil as adulterant were above 15%. However, the accuracy was low when the concentration of adulteration level was below 15%. This study is considerably innovative to drive a new direction of spectroscopy research since a miniature handheld device was used.

Furthermore, quantification and classification of adulterations in olive oil were proposed in another research⁴⁸⁾. Corn and sunflower oil were used as adulterants in the olive oil. It was reported that there was no misclassification error in discriminant analysis used to distinguish between pure EVOO and adulterated with corn oil and sunflower oil. Furthermore, high R^2 value was also obtained to quantify the level of sunflower oil adulterated olive oil samples. PLS was used for calibration of the FTIR spectrum collected during the experiment. The FTIR spectrometer was a tabletop portable type of instrument equipped with Attenuated Total Reflectance (ATR) kit to produce deep penetration in infrared beam of 2 μm .

Another attempt to detect adulteration in olive oil was also proposed with NIR spectroscopy instrument combined with PCA and SIMCA for calibration⁷⁾. Various type of oils was used as adulterants. The PCA scores plot showed separation between the adulterated mixtures and the unadulterated sample, which demonstrated that the developed method could detect as low as 2.7% w/w adulteration if an unadulterated sample of the oil in question was provided. Lower limits of adulteration detection for corn, sunflower, soybean, and canola oils were found to be approximately 20%, 20%, 15%, and 10%, respectively.

While high-resolution nuclear-magnetic resonance (NMR) spectroscopy is an expensive instrumentation and not commonly available, it has grown in popularity with lipid scientists. Fragaki *et al.* reported that three grades of olive oils were able to be classified by combining two multivariate statistical methods, hierarchical cluster analysis and decision analysis, by applying High-field ^{31}P NMR (202.2 MHz) spectroscopy⁴⁹⁾. Agiomyrgianaki *et al.* used NMR spectroscopy for the detection of refined olive oil adulterated with refined hazelnut oil⁵⁰⁾. In this study, the refined oils were classified based on their fatty acids content and the concentration of their minor compounds by using the forward stepwise canonical discriminant analysis and the classification binary trees.

Proton NMR spectroscopy combined with PCA and PLS was further applied for detection of adulteration of camellia oil with three different cheap vegetable oils at different

percentages ranging from 5% to 80%⁵¹⁾. The slight difference between these oils was also demonstrated using a discriminant analysis with an identification accuracy of more than 90% in this research⁵⁰⁾. As the summarizing part of this section, Table 3 shows the research review in the edible oils' adulteration detection using spectroscopy method. Despite the fact that the spectrometric method has been proposed as a simple and fast detection method, it is difficult to achieve complete characterization of a sample's quality³³⁾. Furthermore, while NMR methods have the advantages of being quick and nondestructive analytical methods, their application fields are limited due to the high cost of the instruments³³⁾.

5.3 Electrochemical Sensor method

Electronic nose came on the market decades ago and it is preferred to routine laboratory analysis because it is fast and easy-to-handle⁵²⁾. They are designed to identify and differentiate between complex odours from food samples using a sensor array consisting of widely tuned (non-specific) sensors treated with various chemical or biochemical substances that are sensitive to odours⁵³⁾. The characteristic fingerprint or odour printing will then be generated by an odour stimulus from the group of sensors. These patterns from recognised odours are then used to create a database that is subjected to multivariate analysis in order to identify and classify unknown odours. Metal-oxide gas sensors, metal-oxide semiconductor field effect transistors, acoustic wave gas sensors, electrochemical gas sensors, quartz crystal microbalance sensors, polymer gas sensors, surface acoustic wave devices, field-effect gas sensors, fiber-optic gas sensors and others are all types of electrochemical sensors. They are all available in various types of sensor coating materials, which are categorised according to the additive doping materials, the form and function of the chemical.

Adulteration of EVOO with rapeseed and sunflower oils can also be detected by using electronic nose based on metal oxide sensors (HS-E nose) and by direct coupling of SPME to MS (SPME-MS)⁵⁴⁾. By applying PCA and PLS analysis to the data, excellent results in predicting the adulteration percentage of the samples were achieved in the study.

Electronic nose is also proven to be very efficient in distinguishing between virgin olive oil (VOO) samples adulterated with different percentages of hazelnut oil by combining data analysis of volatile substances by using the PCA⁵⁴⁾. In this research, virgin olive oil (VOO) adulterated with 5 to 50% hazelnut oil was successfully detected by applying this method. Use of surface acoustic wave sensing electronic nose, was able to produce a two-dimensional olfactory image called VaporPrintTM for detection of adulteration in refined, bleached, deodorized (RBD) palm olein with lard.

Table 3 Summary of research in edible oils adulteration detection using spectroscopy method.

Authors	Oil sample	Adulterants	Instrument	Calibration method	
				Quantitative (regression)	Classification
Ozulku <i>et al.</i> ³⁴⁾	Cold-press sesame oil	hazelnut, canola, and sunflower oils	FTIR-ATR spectroscopy	PLS	PCA and Cluster analysis
Rohman and Che Man ⁴⁸⁾	Extra virgin olive oil	Corn oil and sunflower oils	FTIR-ATR spectroscopy	PLS	Discriminant analysis (DA)
Rohman and Che Man ³⁵⁾	Cod-liver oil	Animal fat (lard)	FTIR-ATR spectroscopy	PLS	Discriminant analysis (DA)
Quiñones-Islas <i>et al.</i> ³⁶⁾	Avocado oil	Sunflower, canola and soybean oils	Mid-FTIR spectroscopy	PLS	SIMCA
Uysal <i>et al.</i> ³⁷⁾	Butter	Margarine	Raman spectroscopy	PLS and ANN	PCA
Zhang <i>et al.</i> ³⁸⁾	Extra virgin olive oil	Soybean, corn and sunflower seed oils	Raman spectroscopy	ESM and SVM	N/A
El-Abassy <i>et al.</i> ⁴⁰⁾	Extra virgin olive oil	Sunflower oil	Vis-Raman spectroscopy	PLS	PCA
Ali <i>et al.</i> ⁴¹⁾	Extra virgin olive oil	Sunflower oil	Fluorescence spectroscopy	PLS	PCA
Karuk Elmas <i>et al.</i> ⁴²⁾	cold pressed grape seed oil	Refined soybean oil	synchronous fluorescence spectroscopy	PLS	PCA and SIMCA
Ntakatsane <i>et al.</i> ⁴³⁾	Butterfat	Vegetable oils	Fluorescence spectroscopy	PLS	PCA
Basri <i>et al.</i> ⁴⁵⁾	Palm cooking oil	Lard oil	Portable MicroNIR spectroscopy	PLS with CARS	SIMCA
Picouet <i>et al.</i> ⁴⁶⁾	Sunflower oil	Mineral oils	Quasi Imaging Visible NIR spectrometer	PLS	N/A
Irfan <i>et al.</i> ⁴⁷⁾	Palm cooking oil	Recycled cooking oil	Handheld Micro-NIR spectrometer	N/A	PCA
Vanstone <i>et al.</i> ⁷⁾	Extra virgin olive oil	corn, sunflower, soybean, and canola oils	NIR spectroscopy	N/A	PCA
Agiomyrgianaki <i>et al.</i> ⁵⁰⁾	Olive oil	Refined hazelnut oil	NMR spectroscopy	N/A	canonical DA and classification binary trees (CBTs)
Shi <i>et al.</i> ⁵¹⁾	camellia oil	3 types of vegetable oils	1H Proton NMR spectroscopy	PLS	PCA and orthogonal projection to latent structures DA (OPLS-DA)
Fragaki <i>et al.</i> ⁴⁹⁾	Extra virgin olive oil	lampante olive oil and refined olive oil	NMR spectroscopy	N/A	Discriminant analysis (DA)

Electronic noses can also be used to identify and distinguish lard from other kinds of animal fats as well as from foods containing lard⁵⁵⁾. VaporPrint showed the results obtained when the radial angles of the two-dimensional olfactory image representing the sensor yield individual patterns (smell prints) of the odour of various animal body fats. In Nurjuliana *et al.*, the PCA was applied to analyse the results obtained and resulted in a satisfactory grouping of samples that also enhances the ability to detect the presence of lard in food items that will assist Halal authentication

in the future⁵⁵⁾. Surface acoustic wave sensing electronic nose was used to detect adulteration of virgin coconut oil with refined, bleached and deodorized palm kernel olein at a level of adulteration from 1 to 20% by generating a pattern of volatile compounds present in the samples (Fig. 5)⁵⁶⁾. By utilizing this device helped the research to obtain a fingerprint of volatile compounds occurring in the oil samples. Nonetheless, in this research, PCA was also used to differentiate between adulterated virgin coconut oil with different levels of refined, bleached and

Fig. 5 VaporPrint™ of virgin coconut oil adulterated with different percentages of RBD palm kernel olein⁵⁶⁾. The changes that occurred as palm kernel olein was slowly added to virgin coconut oil were able to be monitored qualitatively by the VaporPrint™ image. This image can be interpreted as the chemical signature of a substance's smell. As virgin coconut oil was slowly adulterated by RBD palm kernel olein, the VaporPrint™ image changed accordingly with the aroma pattern slowly becoming more like the VaporPrint™ of the adulterant as the percentage of adulteration increased.

deodorised palm kernel olein and pure virgin coconut oil, with satisfactory results obtained.

The idea of electronic tongues (E-tongue) or taste sensors has grown rapidly in recent years due to their high potential. Electronic tongues may be viewed as wet alternatives of e-noses. The performance of a non-specific array of sensors reveals various patterns for different taste-causing chemical substances and the resulting data is statistically processed⁵³⁾. The performance pattern is determined by the different selectivity of the individual sensing units and is associated with a particular taste or quality feature. The electronic tongue consisted of a double working electrode (platinum and gold), an auxiliary and a reference electrode onto which both large amplitude pulse voltammetry (LAPV) and small amplitude pulse voltammetry

(SAPV) were applied⁵⁴⁾. It is further developed into a different configuration consisting of five working electrodes (gold, iridium, palladium, platinum and rhodium), a reference electrode and an auxiliary electrode of stainless steel⁵⁷⁾. Electronic tongue has been used for a wide variety of applications, including the detection of adulteration in edible oils due to their robustness and simplicity.

Whether mislabelled or blended with cheaper olive and seed oils, the high price of EVOOs and virgin olive oils (VOOs) make them perfect "candidates" for adulteration. According to Apetrei and Apetrei, the use of voltammetric e-tongue has been shown for the first time to detect adulteration in EVOO with different concentrations of sunflower oil, corn oil and soybean oil (Fig. 6)⁵⁷⁾. In this research, PCA, partial least squares discriminant analysis (PLS-DA)

Fig. 6 Voltammetric responses of voltammetric e-tongue, EO carbon paste-based sensors⁵⁷⁾. Voltammetric measurements were carried out by means of Square wave voltammetry (SWV). Preliminary studies and stabilization of the sensor signals by cyclic voltammetry (CV). EVOO carbon paste-based sensors: (a) CV; (b) SWV. Sf carbon paste-based sensors: (c) CV; (d) SWV. S carbon paste-based sensors: (e) CV; (f) SWV. C carbon paste-based sensors: (g) CV; (h) SWV. The voltammetric curves indicate that the nature and concentration of electroactive compounds found in different edible oils bearing a diversity of electrochemical signals. The voltammetric responses can be used to discriminate edible oils. From the analyse of the voltammetric curves it can be observed that EVOO related curves can be easily distinguished from other oils.

and PLS regression were also applied to e-tongue data showed the capability of this device to discriminate pure oils (an EVOO and three seed oils) according to their botanical origins, to predict total polyphenolic content of binary mixtures of EVOO and seed oils, to classify the adulterated EVOOs when the concentration of adulterant oil was over 5% and to estimate the composition of EVOO and seed oil mixtures within the range 2-25% range. By the combination of the use of a voltammetric e-tongue and an e-nose based on metal oxide semiconductor sensors and pattern recognition technique, argan oil adulterated with different concentrations of sunflower oil can be detected in the work of Bougrini⁵⁸⁾. However, there is a limited selection of commercially available electrochemical sensors on the market. Furthermore, such electrochemical sensors are typically stationary, necessitate specific measurement conditions, and are costly to manufacture and maintain⁵⁹⁾.

6 Conclusion

With fast distribution systems globally, adulterants that are added intentionally in edible oils can cause severe economy and health consequences. The detection of edible oils adulteration is one of the ways to establish food defense, in which food security and resilience can be enhanced. This paper outlined the regulatory compliance for edible oils in some countries and critically reviewed the detection technologies available such as SPME-GC-MS, NIR, NMR, FTIR, electronic nose and tongue. The continued development of new technologies or enhancement of current methods are essential so that food supply chain can be monitored more effectively.

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