



Detection of Adulteration in Coconut Oil and Virgin Coconut Oil Using Advanced Analytical Techniques: A Review

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Received: 27 January 2022 / Accepted: 1 June 2022 / Published online: 13 June 2022
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Abstract

Coconut (CO) and virgin coconut oil (VCO) are popular functional oils owing to their health benefits. The fraudsters intentionally blend cheap and poor-quality vegetable oils with CO and VCO for economic benefits. Adulteration causes safety and health issues for consumers. In this context, there is a need for rapid, accurate, and precise analytical techniques to detect adulteration in CO and VCO. Conventional analytical methods are arduous and lengthy processes using toxic chemicals and involve tedious sample preparation. Thus, food researchers are in search of precise, rapid, and green analytical techniques to detect and quantify the adulteration of CO and VCO. The present review focuses on summarizing all the analytical techniques along with chemometric tools developed in recent times. Various techniques, such as chromatographic, spectroscopic, and thermal techniques coupled with chemometrics, have been successfully applied in this field. The NIR-based spectroscopic technique is a very optimisable and practical method for adulteration detection in CO and VCO. The official methods currently used must be upgraded as they are comparatively less sensitive than the latest methods.

Keywords Virgin coconut oil · Adulteration · Authentication · Analytical techniques · Vibrational spectroscopy · Chromatographic techniques · Chemometrics · Adulterants

Highlights

- Coconut oil (CO) and virgin coconut oil (VCO) are commonly adulterated with cheaper vegetable oils.
- Authentication and adulteration control are critical in coconut oil industry.
- The prevailing official methods for quality control must be upgraded.
- Spectroscopic techniques for oil adulteration detection are discussed.
- Chromatographic and other techniques for VCO adulteration detection are discussed.

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Abbreviations

VCO	Virgin coconut oil
CO	Coconut oil
RBD	Refined, bleached, and deodorised
ATR	Attenuated total reflectance
FTIR	Fourier transform infrared spectroscopy
FT-MIR	Fourier transform mid-infrared spectroscopy
NIR	Near infrared spectroscopy
PLS	Partial least square
PLS-R	Partial least square-regression
PLS-DA	Partial least square-discriminant analysis
RMSECV	Root mean square error of cross-validation
RMSEP	Root mean square error of prediction
DA	Discriminant analysis
PC	Principal components
SEC	Standard error of calibration
SEP	Standard error of prediction
PCR	Principal component regression
HCA	Hierarchical cluster analysis
LDA	Linear discriminant analysis
DD-SIMCA	Data-driven-soft independent modelling of class analogy

NMR	Nuclear magnetic resonance spectroscopy
PL	Photoluminescence
MCR-ALS	Multivariate curve resolution-alternating least squares
UV-VIS	Ultraviolet-visible spectroscopy
CIE	International Illuminating Committee
INEPT	Insensitive nuclei enhanced by polarisation transfer
MS	Mass spectrometry
MALDI	Matrix-assisted laser desorption/ionisation
TAG	Triacylglycerol
ESI-MS	Electrospray ionisation mass spectrometry
HPLC	High-performance liquid chromatography
FAME	Fatty acid methyl esters
RP-HPLC	Reverse phase high-performance liquid chromatography
GC	Gas chromatography
OPLSDA	Orthogonal projections to latent structures-discriminant analysis
TOF-MS	Time-of-flight mass spectrometry
DSC	Differential scanning calorimetry
SBO	Soybean oil
PKO	Palm kernel oil
SMLR	Stepwise multiple linear regression
E-NOSE	Electronic nose
GC-SAW	Gas chromatography-surface acoustic wave sensor
QDA	Quadratic discriminant analysis
RDA	Redundancy analysis
K-NN	<i>K</i> -Nearest neighbours algorithm
ANN	Artificial neural networks
SVMS	Support vector machine

Introduction

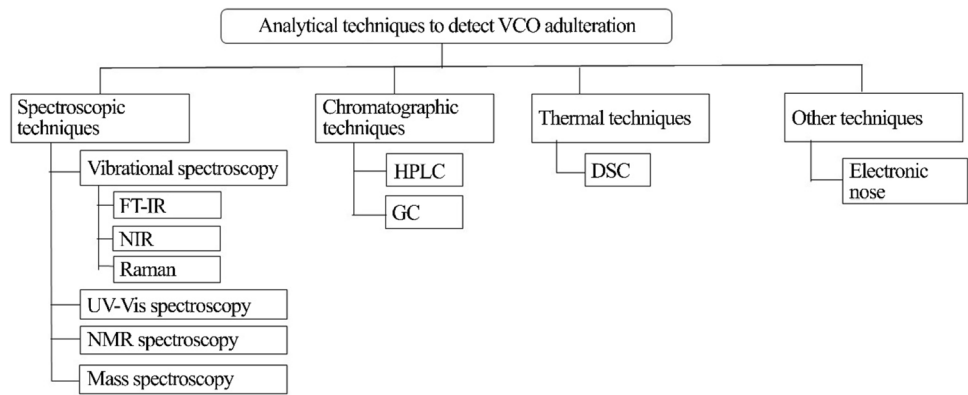
Coconut (*Cocos nucifera*) is a prevalent plantation crop cultivated in more than 80 countries. Coconut oil (CO) is immensely used for food purposes (Rohman et al. 2021). CO is extracted by either dry or wet processing. Dry processing involves pressing ground and steamed copra to extract oil, which then undergoes the processes of refining, bleaching, and deodorizing (RBD). Since dry processing involves heat, CO extracted from cream obtained from coconut milk is called wet processing. Cold-pressed oil processing, the oil-seeds are pressed at room temperature, so the temperature of the oil and acid value are very low, whereas in hot-pressed oil extraction, the oil seeds are subjected to roasting and then pressed through a screw press under high pressure and temperature. The resulting coconut oil is called virgin coconut oil (VCO) (Marina et al. 2009a, b; Manikantan et al. 2018). This process has become more popular as it does not involve any chemical treatment.

Coconut oil is rich in medium-chain fatty acids, including caproic, caprylic, and capric acids, and shows good digestibility. Also, it exhibits antifungal, antiviral, hepatoprotective, cardioprotective, hypolipidemic, and antidiabetic characteristics which are highly beneficial to human health (Salian and Shetty 2018). This contributes to the extremely good quality and high commercial value of the oil. This makes the oil highly susceptible to intentional adulteration with low-cost, low-quality vegetable oils such as palm oil, sunflower oil, corn oil, canola oil, and soybean oil (Neves and Poppi 2020; Pizzo et al. 2019a; Pandiselvam et al. 2015) and other oils (Amit et al. 2020b; Pandiselvam et al. 2015) for economic reasons. In India, there are 14 brands of coconut oil banned by the Food Safety and Standards Authority of India (FSSAI) due to adulteration (Neo 2019). This adulteration not only causes consumer disappointment, but also safety and health issues (Bansal et al. 2017). Oil millers are getting badly affected due to the trading of adulterated coconut oil. In a report, it was noted that 90% of the oil that is traded as coconut oil is palm kernel oil (Keshavaram 2015). In China, low-cost animal fats produced from waste tissues and valueless skin from animal or poultry slaughterhouses were used as adulterants in VCO and peanut oil (Xu et al. 2015).

The European Union Commission and Food and Agricultural Organization have established regulations and quality control methods for oil. However, certain established official methods for the quality control of oil are complicated, laborious, use toxic and expensive chemicals, and require tedious sample preparation procedures (Valli et al. 2016; Rifna et al. 2022). There are two common methods to detect and quantify adulterants in CO and VCO that have been reported, viz, spectroscopic techniques (Amit et al. 2020c; Pizzo et al. 2019a; Popa et al. 2020) and chromatographic techniques (Komaram et al. 2021; Xu et al. 2015) coupled with chemometric tools such as principal component analysis, partial least square regression, discriminant analysis, and clustering analysis (Naveen et al. 2022). Therefore, authentication and adulteration control are critical in the VCO industry.

To the best of our knowledge, there is no review paper on the aspect of advanced analytical techniques application to VCO adulteration detection. This review highlighted the methods of determining adulteration and quantification of VCO along with the statistical analysis (Fig. 1). Different analytical techniques such as spectroscopic techniques (FTIR spectroscopy, NIR spectroscopy, Raman spectroscopy, ultraviolet–visible spectroscopy, mass spectrometry, nuclear magnetic spectroscopy), chromatographic techniques (gas chromatography and high-performance liquid chromatography), thermal techniques (differential scanning calorimetry), and other techniques (electronic nose) have been applied for the identification and quantification of VCO adulteration to date. The application of the E-nose technique

Fig. 1 Various analytical techniques to detect VCO adulteration



for the detection of adulteration is mainly focused. All of these techniques are employed for authentication. Adulteration detection in oil is discussed in the succeeding sections.

Vibrational Spectroscopy for the Determination of Adulteration in CO and VCO

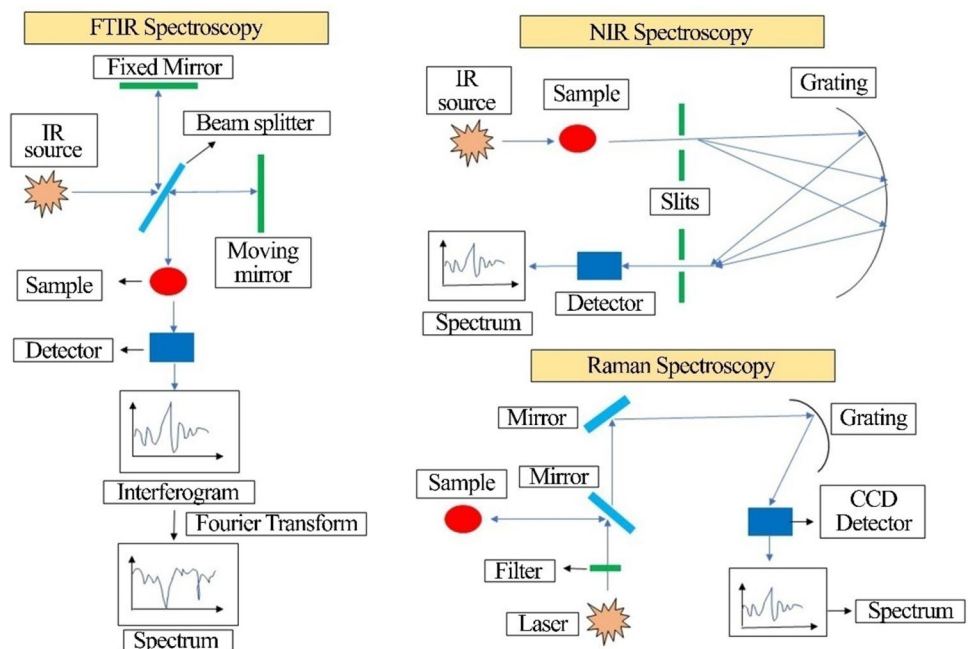
Vibrational spectroscopy assesses the interaction between electromagnetic radiation and the vibrational states of the atomic nuclei within their corresponding molecules. Vibrational spectroscopic techniques have various advantages over other analytical techniques, such as non-destructive, rapid analysis, no tedious sample preparation, and low running costs (Meenu and Xu 2019; Pandiselvam et al. 2021, 2022; Mahanti et al. 2022). Vibrational

spectroscopy encompasses Raman and infrared (near, mid, or far) spectroscopy. Vibrational spectroscopy, in association with chemometrics, has been comprehensively utilised in the food industry (Pandiselvam et al. 2015; Galvin-King et al. 2021; Taylan et al. 2021; Kaavya et al. 2020; Mahanti et al. 2022). Several works have conveyed the application of Raman and infrared spectroscopic techniques for determining and quantifying the adulterants present in coconut oil, which are discussed in the following sections.

Determination of Adulteration by FTIR Spectroscopy

In FTIR spectroscopy (Fig. 2), multiple frequencies can be measured simultaneously by interferometric modulation of radiation in the range of 4000 to 400 cm⁻¹ wavenumber to identify and quantify food constituents (Valand et al. 2020; Rifna et al. 2022). Manaf et al. (2007) conjugated FTIR with

Fig. 2 Schematic diagram of (a) FTIR spectroscopy and (b) NIR spectroscopy



attenuated total reflectance (ATR) and chemometrics (partial least square regression — PLS) to detect the adulterant (palm kernel olein) in virgin coconut oil (VCO). Fourteen sets of pure VCO and eighteen sets of adulterated VCO with palm kernel oil up to 50% by volume were used for data collection using FTIR. The PLS calibration model developed with the actual adulteration concentration versus the predicted adulteration concentration resulted in a high coefficient of determination (R^2) value of 0.9973 and the cross-validation yielded a 0.9875 R^2 value and 1.70 RMSECV value. The discriminant analysis (DA) using 10 principal components (PC) factors was carried out to classify the pure and adulterated samples which resulted in 100% classification with not even a single sample being mistakenly classified. In a similar study conducted by Rohman and Man (2009), FTIR combined with ATR and PLS was employed to detect and quantify adulterated palm oil in VCO. Twenty-five sets of VCO samples adulterated using a palm oil combination of about 1–50% by weight in the presence of chloroform were used for developing the PLS calibration model. Forty independent samples were used for the calibration of the model. APLS calibration model was established using the spectral regions to determine the level of adulteration that showed a linear relationship. The variations between the actual and FTIR-predicted values were described using the R^2 value (0.999) and the SEC value (0.533). The cross-validation resulted in R^2 and standard error of prediction (SEP) values of 0.996 and 0.993, respectively. They were also able to successfully classify pure and adulterated coconut oil with 100% accuracy by employing discriminant analysis using 7 principal component factors, which showed that the developed model can predict the adulteration of up to a 1% lower control limit.

Abdul Rohman and Man (2011) attempted to quantify VCO in the ternary mixture with olive oil and palm oil utilizing FTIR-ATR combined with PLS and principal component regression (PCR). In the spectrum obtained from the ternary oil mixture, there were bands observed for olive oil and palm oil, but no bands were found for VCO at the frequencies of 3005 and 1654 cm^{-1} . Such differences have been exploited to determine the VCO content in complex oil mixtures. The FTIR spectral data was pre-processed by the mean centering method, then PCR and PLS models were built based on the original and derivative (first or second) spectra at 1200–1000 cm^{-1} frequency regions for VCO quantification. The PLS model constructed using the second derivative spectra reveals the highest R^2 (0.9999) value and the lowest RMSEC (0.200) value. In an identical study, FTIR-ATR with multivariate calibration methods (PLS and PCR) has been applied to find VCO content in binary oil mixtures (VCO and olive oil; VCO and palm oil) (Rohman et al. 2010). The distinction between the VCO and olive oil spectrum was found at 1111 cm^{-1} and 962 cm^{-1} frequency regions, whereas the distinction between the

VCO and palm oil spectrum was found at 1120–1105 cm^{-1} and 965–960 cm^{-1} frequency regions. The PLS calibration models constructed at these spectral frequency regions were reported to be the best in terms of the highest R^2 (0.9992) and lowest RMSEC (0.756) for the binary mixture of VCO and olive oil. Meanwhile, the R^2 and RMSEC values of the binary mixture of VCO and palm oil were found to be 0.996 and 0.494 respectively, proving FTIR along with PLS to be one of the efficient techniques to quantify VCO in oil mixtures.

The virgin coconut oil adulterated with sunflower oil and corn oil was determined using FT-MIR spectroscopy in conjunction with chemometrics (PLS and DA) (Rohman and Che Man 2011). DA using 10 PCs was able to classify pure and adulterated oil samples with 100% accuracy. The adulterants which were successfully quantified using the PLS regression model resulted in the highest R^2 (0.999) value and low RMSEP values for both adulterants. In the same way, lard adulteration in VCO has been detected and quantified by employing FTIR spectroscopy coupled with chemometrics (Tengku Salwani, Tengku Mansor et al. 2011). The frequency regions selected for PLS and DA analysis were 3020–3000 cm^{-1} and 1120–1000 cm^{-1} . The established PLS model was able to effectively predict lard content in VCO with a high R^2 (0.9970) value and RMSEC of 1.54%. Also, the classification of pure VCO and lard adulterated samples were conducted by DA using 6 PC factors and none of the samples was misclassified. The VCO adulterated with mustard and palm oil was detected by FTIR with the assistance of hierarchical cluster analysis (HCA), principal component analysis (PCA), and DA (Mk et al. 2017). Similarly, ATR-FTIR has been reported to analyse the adulteration of VCO with mustard oil with the help of chemometric tools (Amit et al. 2020a). The LDA revealed 100% exact classification between pure and adulterated samples, and the selected spectral frequency regions for PCA analysis ranged from 3010 to 2800 cm^{-1} and from 1800 to 720 cm^{-1} . The PLS-R and PCR models were constructed based on the original and derivative spectra in the same frequency region. The PLS-R model showed excellent prediction with the highest R^2 of 0.999 and the lowest limit of detection of mustard oil in VCO being 1% v/v.

Paraffin oil is one of the major adulterants of VCO, which was identified by the ATR-FTIR technique associated with some chemometric tools. PCA and linear discriminant analysis (LDA) efficiently (100%) distinguished adulterated samples from pure VCO. The PLS-R model yielded the highest coefficient of determination ($R^2 = 0.999$, RMSEC = 0.142, and RMSEP 0.162) with the lowest limit of detection of paraffin oil in VCO being 1% v/v (Amit et al., 2020b). VCO is not only adulterated with cheap vegetable oils but also adulterated with fried coconut oil and this adulteration can be determined by ATR-FTIR spectroscopy (Amit et al. 2020c).

The spectral frequency regions selected for PCA were 3000 to 2800 cm^{-1} and 1800 to 500 cm^{-1} and the PCA 3D plot excellently separated the pure and adulterated samples into different clusters. The LDA exhibited 100% exact classification between pure and adulterated samples and the lowest detectable limit of fried coconut oil in pure coconut oil was found to be 0.5% v/v. The PCR and PLS-R models were built using original and derived spectra in which the PLS model for the first derivative at the frequency regions 1800 to 500 cm^{-1} was reported to have predicted the best with high accuracy.

The ATR-FTIR spectroscopy, in tandem with DD-SIMCA (data-driven-soft independent modelling of class analogy) has been utilised to assess the authenticity and adulteration of VCO which is adulterated with corn oil, canola oil, sunflower oil, and soybean oil (Neves and Poppi 2020). The PCA was applied to all the spectra of pure and adulterated coconut oil, and a clear separation was found between them, showing that the proposed methodology was able to verify the authenticity of coconut oil. The developed DD-SIMCA models to identify vegetable oil adulterants showed acceptable results, with high sensitivity (88.23–100%) and specificity (95.74–100%) in the prediction step. FTIR can be used to detect and quantify adulteration. However, the Fourier transform (FT-NIR) spectroscopy records the interferogram based on time-domain measurements.

Determination of Adulteration by NIR Spectroscopy

Near infrared (NIR) spectroscopy (Fig. 2) utilises the NIR region (800 to 2200 nm) of the electromagnetic spectrum to measure the molecular overtones and combination bands of a few fundamental vibrations (Ozaki et al. 2021; Kaavya et al. 2020; Pandiselvam et al. 2022). The adulteration of palm olein in VCO was detected and quantified by FT-NIR using a PLS model (Jayatunga et al. 2020). For the analysis, 20 VCO samples with adulteration ranging from 1 to 70% v/v of palm olein in CCO were prepared by vortex at 1500 rpm for 2 min. A PLS model was developed and validated for the prediction of adulteration with the regression coefficient (R^2) of 0.9997 and 0.9993. There is a high correlation between PLS calculated and actual values of adulteration. NIR spectroscopy has been applied to determine adulteration in edible oils, for instance, coconut oil which is adulterated with paraffin oil has been detected in a study (Raj et al. 2018). The samples are prepared with 0, 1, 2, 3, 4, 5, 10, and 100% of paraffin oil which are labelled as P0, P1, P2, P3, P4, P5, P10, and P100, respectively. The peaks appearing in the NIR spectrum (1200–1800 nm) of pure paraffin oil indicate the vibration of an alkyl group whereas the coconut oil spectrum shows additional peaks at 1904, 1928, and 2132 nm corresponding to the vibration of the carbonyl group. At 2132 nm, the intensity of the peak

decreased with a decrease in paraffin oil content, and thereby the adulterant content was quantified with the highest R^2 value of 0.986. NIR spectroscopy works in the spectrum of 750- to 2600-nm electromagnetic region. C-H, O-H, and N-H bonds molecules in the samples absorb NIR waves at specific wavelengths. These absorbances are and the difference in the absorbance is used to predict adulteration.

Determination of Adulteration by Raman Spectroscopy

Raman spectroscopic technique provides distinctive fundamental vibrations that can be used to elucidate the sample's molecular structure (Bhunia et al. 2014). Raman spectroscopy, in conjugation with multivariate curve resolution-alternating least squares (MCR-ALS) was utilised for the detection of adulteration of coconut oil contaminated with other relatively cheap oils such as palm kernel, castor bean, peanut, soybean, sunflower, canola, sesame, mineral, babassu, and Vaseline oils. The acquisition of spectra was in the range of 3200 to 200 cm^{-1} and at 2 cm^{-1} resolution. The control charts were built using the score values from MCR-ALS analysis, and they were reported to successfully differentiate adulterated samples (adulteration level — above 2%) from the pure ones. The adulterant content was quantified using regression models constructed using the MCR-ALS algorithm, and it showed excellent prediction results (De Géa Neves and Poppi 2018). The list of the utilisation of vibrational spectroscopic techniques for the detection and quantification of adulteration in virgin coconut oil is summarised and presented in Table 1.

Other Spectroscopic Techniques for the Determination of Adulteration in CO and VCO

Determination of Adulteration by Ultraviolet-Visible Spectroscopy

The cold-pressed coconut oil adulterated with its refined versions was reported to be detected rapidly using UV-Vis spectroscopy (Popa et al. 2020). They have also employed FTIR spectroscopy to analyse its ability to establish adulteration of cold-pressed coconut oil with refined coconut oil and observed some notable differences in the spectra between cold-pressed (pure) and adulterated ones, but it is not enough to quantitatively determine the saturated and unsaturated fatty acid content, whereas the colour analysis by the UV-Vis spectroscopy technique was found to be a promising method for distinguishing the pure cold-pressed coconut oil from its adulterated refined version. The results of

Table 1 List of vibrational spectroscopic techniques for determination of adulteration in CO and VCO

Adulterants	No. of samples	Spectroscopy technique	Range of spectrum, cm^{-1}	Resolution, cm^{-1}	Chemometrics	Limit of detection	References
Palm kernel olein	41	FTIR	4,000 to 650	4	PLS, DA	1%	(Manaf et al. 2007)
Palm oil	125	ATR-FTIR	4,000 to 650	4	PLS, DA	1%	(Rohman and Man 2009)
Olive oil	65	ATR-FTIR	4,000 to 650	4	PLS, PCR	1%	(Rohman et al. 2010)
Palm oil	65	ATR-FTIR	4,000 to 650	4	PLS, PCR	1%	(Rohman et al. 2010)
Palm oil and olive oil	24	ATR-FTIR	4,000 to 650	4	PLS, PCR	1%	(Rohman and Man 2011)
Corn oil and sunflower oil	80	ATR-FTIR	4,000 to 650	4	DA, PLS	2%	(Rohman and Man 2011)
Lard	30	ATR-FTIR	4,000 to 650	4	DA, PLS	1%	(Mansor et al. 2011)
Mustard oil and palm oil	7	ATR-FTIR	4,000 to 650	4	HCA, PCA, DA	5%	(Mk et al. 2017)
Paraffin oil	128	ATR-FTIR	4,000 to 400	4	PCA, LDA	1%	(Amit et al., 2020b)
Fried coconut oil	83	ATR-FTIR	4,000 to 400	4	PCA, LDA, PLS	0.5%	(Amit et al. 2020c)
Canola, corn, sunflower and soybean oil	123	ATR-FTIR	4,000 to 400	4	PCA, DD-SIMCA	-	(Neves and Poppi 2020)
Mustard oil	120	ATR-FTIR	4,000 to 400	4	PCA, LDA	1%	(Amit et al. 2020a)
Paraffin oil	32	NIR	4000 to 200	4	-	2%	(Raj et al. 2018)
Palm kernel, castor bean, peanut, soybean, sunflower, canola, sesame, mineral, babassu, and Vaseline oils	150	Raman	3200 to 200	2	MCR-ALS, PCA	1%	(De Géa Neves and Poppi 2018)

colour analysis show that there is a difference in the absorption spectra and the CIE (International Illuminating Committee) L^* , a^* , and b^* values between cold-pressed oil and the refined one. The parameter a^* and b^* values were found to be three and six times higher, respectively, for refined coconut oil than for cold pressed coconut oil. The parameters a^* and b^* were reported to be linearly dependent with the highest correlation coefficient ($R^2 = 0.99$) and from which the cold-pressed coconut oil content in a given sample can be determined. Thus, the only UV–Vis spectroscopic study by Popa et al. (2020) proved to be an excellent method to detect virgin coconut oil adulteration. A similar kind of work has been done for the detection of adulteration in virgin olive oil using UV–VIS. The EVOO and mixed olive oil were subjected to UV analysis at 232 to 400 nm by a spectrophotometer (Jen Way 6105 UV–VIS, Esses, England). The samples were heated for 45 and 90 min to assess the thermal degradation. The samples were prepared by diluting 250 mg of the oil samples in iso-octane solution and diluted to a 0.8 mg/mL concentration. The adulterated EVOO with mixed oils highly absorbed the UV–VIS light between the 240- and 300-nm band and the study revealed that the UV–VIS spectrometer is the most suitable and reliable technique to detect and

quantify high levels (over 10%) of adulteration in the EVO (Gholami Khesht et al. 2020).

Determination of Adulteration by Nuclear Magnetic Resonance Spectroscopy

The ^{13}C INEPT NMR spectroscopy, along with chemometrics, with an untargeted finger printing approach, has been employed for the authentication of numerous commercial food products by using a spectral database built with 294 spectra of raw plant and animal fats and oils (Guyader et al. 2018). The plant oil (including coconut oil) belonging to one origin was spiked with another and the limit of detection was found to be around 5%. Also, when animal oil was spiked with plant oil, the limit of detection was 2%. These limits cannot be achieved by a fatty acid profile. Even though spectroscopic methods like FTIR can reach these limits, this method was proved to cover a vast range of oils of plant and animal origins. The PCA analysis was able to discriminate between plant and animal oils and among 30 origins of plant oils. ^{13}C NMR was also employed to distinguish between VCO, degraded VCO, adulterated VCO, and RBD coconut oil with the help of untargeted multivariate analysis (Lagurin et al. 2020). The PCA analysis was able to differentiate among all the coconut oil samples. The PLS-DA was used as a binary classifier, and

the optimised model resulted in improved performance in differentiating all the coconut oil samples.

Determination of Adulteration by Mass Spectroscopy

The MALDI-MS, matrix-assisted laser desorption/ionisation mass spectrometry, was used to create a complete spectral database which includes more than 30 edible oil species (including coconut oil), for the quick cataloguing of edible oil samples by comparing them with the reference MALDI-MS spectra in the database (Ng et al. 2018). Many research have shown that the triacylglyceride (TAG) region in the MALDI-MS spectra of different edible oils was specific which can be useful for the description and cataloguing of edible oils (Ng et al. 2015). The TAG region of coconut oil was dominated by peaks less than m/z 850, with abundant peaks in the lower mass region (m/z 689.6) corresponding to TAGs with C14:0. This specific TAG was only found in coconut oil, which made it cluster separately from the rest of the oils in the PCA plot, and the established PLS-DA models accurately classified 97.2% of all the edible oils.

Coconut oil adulterated with some cheaper quality soybean oil has been detected using direct infusion electrospray ionisation mass spectrometry (ESI-MS) (Pizzo et al. 2019b). The lipid profile spectra were obtained and the $[TAG + NH_4]^+$ of the region 870.9 m/z was selected as the lipid marker of soybean oil, which is absent in coconut oil. A calibration curve was built using $[LLnP + NH_4]^+$ intensity versus soybean oil percentage to effectively quantify adulterant content with the highest R^2 of 0.994 and the lowest limit of detection is 2%. In a similar study, direct infusion ESI-MS combined with chemometrics was applied to detect

coconut oil adulterated with palm kernel oil (Pizzo et al. 2019b). The lipid profile spectra of coconut oil and palm kernel oil were obtained in the region of 100–1200 m/z by direct infusion (ESI-MS). The principal component analysis of ESI-MS data shows that it separated adulterated samples from pure ones, indicating that mass spectrometry can effectively detect adulteration in coconut oil.

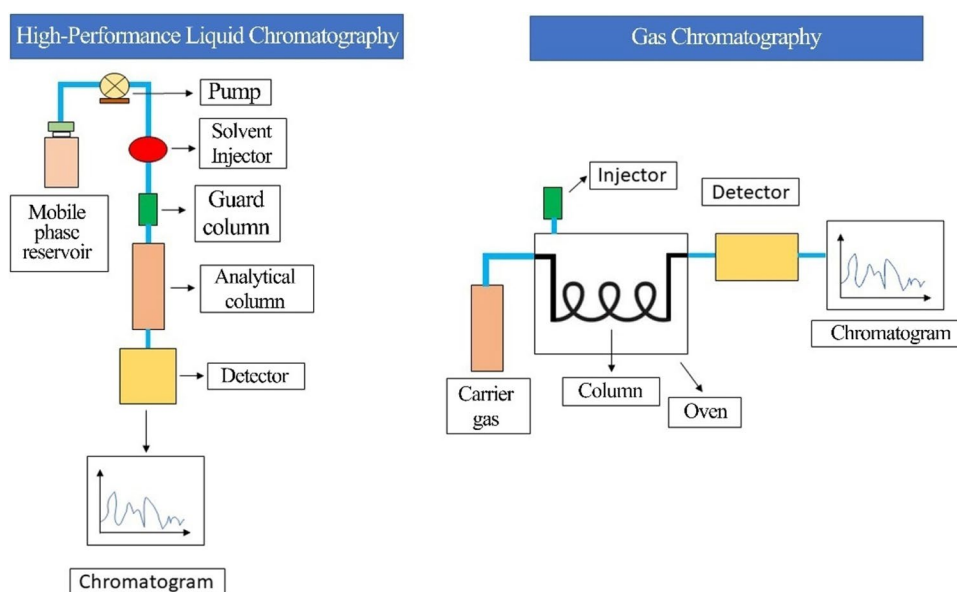
Chromatographic Techniques for the Determination of Adulteration in CO and VCO

The chromatographic techniques (Fig. 3) used for the detection of coconut oil adulteration are based on specific marker compound quantification. The tocopherols, triglycerides, tocotrienols, phenolic compounds, polar fraction, and pyropheophytins are the marker compounds that can be determined by HPLC, whereas the fatty acid methyl esters (FAME), sterols (stigmasterol and campesterol), stigmastadiene, triacylglycerol, and volatile compounds are the marker compounds that can be determined by gas chromatography.

Determination of Adulteration by High-Performance Liquid Chromatography

The coconut oil adulterated with palm olein and palm kernel oil has been determined using a reverse phase (RP)-HPLC-based method using triglyceride as a marker (Komaram et al. 2021). The three major triacylglyceride molecular species of coconut oil are trilaurin (C36), dilaurin-monocaprin/myristin-caprylin-laurin (C34), and dilaurin-monomyristin (C38) from which trilaurin was

Fig. 3 Schematic diagram of (a) HPLC and (b) GC



selected as a detection index for quantification of the level of palm kernel oil. Whereas to quantify palm oil adulterant in coconut oil, the ratio of peak area of dipalmitoyl-monoolein and trilaurin was chosen as detection index. The developed RP-HPLC based method proves to be satisfactory with 2–4% lowest limit of detection and detection accuracy being 78–98%, varying with the type of oil and level of adulteration. Information about the HPLC method for authentication of CO and VCO was acute, so the use of the HPLC method for detecting adulteration in other oils was discussed. By estimating the phenols and tocopherols in virgin olive oil, HPLC was used to differentiate the oil quality. Simultaneous extraction of virgin olive oil was carried out using methanol and isopropanol-methanol mixture and the extract was separated by HPLC using a reversed phase C18 column with acetic acid/water–methanol-acetonitrile-isopropanol mixture under gradient elution methods (Tasioula-Margari and Okogeri 2001). Similarly, in another study, triglycerides of extra/virgin olive oil tainted with 5% soybean oil were determined by HPLC for adulteration detection (Jabeur et al. 2014). The extra-virgin olive oil was adulterated with the soybean, sunflower, and corn oils at the levels of 1, 2, 3, 4, 5, and 10% w/w which were separated using an ion trap mass spectrometer interfaced to the HPLC system through an atmospheric pressure chemical ionisation (APCI) (Fasciotti and Netto 2010). The study reported that the TAGs were separated at isocratic elution with a 60:40 acetone/acetonitrile mixture; 137 column temperature, 30 °C; flow rate, 1.5 mL/min; and injection volume of 20 µL of the sample and identified by their mass and equivalent carbon number (ECN). The absolute difference between theoretical and experimental ECN42 content (ΔECN_{42}) in all seed oils was higher than in extra-virgin olive oil. The ΔECN_{42} value must not exceed 0.20 in the case of edible virgin olive oil, 0.30 in the case of lampante olive oil, 0.50 in the case of olive pomace oil, and 0.60 in the case of crude olive pomace.

Extra virgin olive oil (EVOO) and a commercial oil blend labelled as 15% (w/w) of olive oil in soybean oil were analysed by HPLC using a reverse-phase LiChrospher octadecyl (ODS) column (250 mm × 3 mm; 5 µm) and a guard column of similar characteristics (Vydac ODS, 201TP54, 2.1 mm; 10 µm). The analysis conditions were a constant flow rate of 0.7 mLmin⁻¹, the column temperature of 35 °C, and the injected volume of 5 µL.

Determination of Adulteration by Gas Chromatography

Gas chromatography mass spectrometry (GC–MS) and NMR fingerprinting techniques in conjugation with chemometrics

were employed for the classification of different types of oil, including coconut oil (Fang et al. 2013). The PCA models of both techniques were able to cluster 14 different types of oils. The PLS-DA and orthogonal projections to latent structures — discriminant analysis (OPLS-DA) models built using GC–MS data resulted in better classification specificity and sensitivity in comparison with models built using NMR data. However, data from either of the two techniques can be efficiently applied to establish an oil and fat database for the identification of unknown samples.

A new method was developed to identify the presence of animal fats in VCO, with cholesterol as a marker, employing two-dimensional gas chromatography (GC × GC) conjugated with time-of-flight mass spectrometry (TOF–MS). The better baseline separation of trimethylsilyl ether (sterol derived from cholestanol and cholesterol) was obtained from GC × GC systems compared to one-dimensional GC so that the adulterant content could be precisely determined. This method can identify the presence of chicken fat, beef tallow, mutton tallow, lard, or their blends in VCO, with a detection limit as low as 0.25% (Xu et al. 2015). GC system can be used to detect and quantify adulteration in VCO by developing and validating an appropriate method.

Thermal Analysis Technique for the Determination of Adulteration in CO and VCO

Determination of Adulteration by Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is a thermo-analytical technique that elucidates the temperature and heat flow related to the transition of materials as a function of temperature and time (Farah et al. 2018; Mahanti et al., 2021). The heating and cooling thermograms of DSC were used to determine adulteration in VCO with soybean oil (SBO) and palm kernel oil (PKO) (Marina, et al. 2009a, b). The melting and crystallisation enthalpy of VCO adulterated with SBO and PKO were studied. The SBO-adulterated sample's heating curve revealed that the adulterant peak occurs at the low-temperature region at an adulteration level of 10%. While the PKO-adulterated samples did not show any adulterant peak, a gradual decrease in peak height was observed in the major exothermic peak, the stepwise multiple linear regression was applied to quantify adulterant content with a high R^2 value of 0.9490.

The thermal profiling of coconut oil employing DSC can be useful for the detection of lard adulteration in it. In the heating thermogram of the adulterated sample, with increasing lard content, one major endothermic peak and a minor peak, which is

progressively smoothening out to become a major peak, were observed. Whereas one minor peak and two major exothermic peaks were observed in the cooling thermogram, one peak increased and the other decreased with lard content. The SMLR analysis shows that two independent variables were able to quantify lard content with R^2 of 0.9582 (Mansor et al. 2012). In another study, they detected adulterated palm olein in VCO by DSC. There was a significant difference between the heating and cooling curves of VCO and palm olein. The changes in the thermal transitions were due to the varying ratios of saturated to unsaturated triacylglyceride molecules. The detected adulterant peaks were correlated with the degree of adulteration using SMLR and resulted in a high correlation coefficient and very low standard error values (Marikkar 2019). Studies indicate that adulteration is altering the glass transition temperature (T_g) by changing the heat capacity and viscosity of oil based on the oxidation of oils and other chemical reactions happening in the VCO.

Recent Techniques for the Determination of Adulteration in CO and VCO

Determination of Adulteration by Electronic Nose

The E-nose can reproduce human senses to artificially perceive volatiles and distinguish them using an array of sensors and pattern recognition systems. Lately, electronic nose systems along with chemometrics are being evolved as a rapid and reliable technique for the detection of adulteration of edible oils (Roy and Yadav 2021). Fast gas chromatography-surface acoustic wave sensors (GC-SAW E-nose) were employed to detect coconut oil adulteration with palm olein as an adulterant. From the chromatographic profile, the adulterant peak was provisionally identified as methyl dodecanoate and its intensity was correlated (using PLS analysis) with the level of adulterant added ($R^2 = 0.91$). The Pearson's correlation coefficient obtained between the adulterant peak and the chemical analysis, viz., iodine value, and peroxide value were 0.92 and 0.89, respectively. Also, the PCA resulted in excellent discrimination between pure and adulterated coconut oil samples (Marina et al. 2010). In a similar study, the GC-SAW E-nose system was employed to identify lard adulteration in VCO. The chromatographic profiles identified ten different adulterant peaks, out of which one peak had the best relationship with lard content in VCO having the highest R^2 value of 0.934 (Tengku Salwani Tengku Mansor et al. 2011) proving E-nose to be an efficient method for the identification of coconut oil adulteration (Table 2).

Chemometrics

Chemometrics, a chemical discipline, uses mathematical statistics, information technology, and probability theory to analyse multidimensional chemical data. The overview of commonly used chemometric tools is summarised in Table 3. Traditional statistical methods are often times not sufficient for precise interpretation of results and can only analyse single variables, whereas chemometrics enables us to obtain valuable information from a broad range of intricate datasets and allows us to detect the relationships between the variables (Esteki et al. 2018). The key advantages of chemometric methods are that they lower the number of measurements and analyses required, reduce costs, and save time. Therefore, chemometric tools, especially multivariate analysis, find numerous applications in food quality control, food safety, and qualitative and quantitative determination of chemical parameters to evaluate food authenticity and adulteration (Gómez-Caravaca et al. 2016; Kaavya et al. 2020; Pandiselvam et al. 2022; Rifna et al. 2022).

Chemometric techniques of pattern recognition are divided into unsupervised pattern recognition and supervised pattern recognition. Unsupervised pattern recognition can determine the structure of a dataset based on its measurements. It displays clusters with respect to the type and number of classes. The results are revealed as two- or three-dimensional graphs or dendograms. The most popular unsupervised pattern recognition techniques are CA, HCA, and PCA. In supervised pattern recognition, object membership in the classes and the number of classes are known beforehand. The unknown samples can be projected into known classes based on experimental results. Supervised pattern recognition may include QDA, LDA, RDA, SIMCA, K-NN, PLS-R, ANN, and SVM methods (Efenberger-Szmechtyk et al. 2018). As previously reported, several research studies have adapted the chemometric tools in association with analytical techniques like chromatography and spectroscopy to identify food scandals, adulteration, and the authenticity of virgin coconut oil.

Limitations of Advanced Analytical Method

Chromatography techniques are physical separation methods to determine and identify the various compounds based on their density by making the mobile phase move on the surface of the stationary phase. Some errors occur during chromatography, such as improper sample loading, incubation temperature and flow rate of the samples, and sample preparation methods. Solvent and standard requirements are one of the major limitations of chromatography techniques. They are very costly and very difficult to obtain for each sample. All the chromatography techniques require trained

Table 2 Advantages and disadvantages of different analytical techniques

Analytical technique	Advantages	Disadvantages
Infrared spectroscopy	Minimum sample preparation High reproducibility Non-destructive Rapid method	Data analysis and building calibration models are tedious and time consuming
Raman spectroscopy	Minimum sample preparation Minimum sample requirement Rapid method Non-destructive Analysis of packaged sample is possible	Thermal degradation of the sample can happen due to high intensity laser Adulterant or fluorescence of sample can interfere with spectra
Nuclear magnetic resonance spectroscopy	Minimum sample preparation Non-destructive Rapid method	Expensive Low sensitivity Large amount to sample is needed
Differential scanning calorimetry	User-friendly Environment-friendly Rapid method	Suitable only for qualitative analysis Sample preparation is time consuming Detecting adulteration in high oleic vegetable oils is impossible by deconvolution analysis
High-performance liquid chromatography	Highly precise High reproducibility High resolution	Expensive Sample preparation is tedious and time consuming Developing new methods are laborious Trouble shooting a problem is difficult
Gas chromatography	High efficiency High sensitivity Precise and accurate Rapid method	Sample preparation is time consuming

manpower to operate. The chromatography instruments are so expensive and handling is very difficult. The operating costs and power requirements are also the major disadvantages of chromatography techniques. Troubleshooting procedure and validation errors, measurement errors that result in inaccurate readings due to parallax errors, transcription errors, and conversion and calculation errors are all barriers to using analytical methods.

The limitations of the chromatography techniques can be overcome by the spectroscopic techniques, since the spectroscopic techniques are non-destructive and allow the use of the sample matrix as such without any destruction. The samples used in spectroscopic techniques can be used for further analysis. So, sample error will be reduced. Spectroscopic measurements are easy and quick. Handling the instruments is also easy and requires less trained personnel. However, the selection of wavelength and light scattering through samples cause serious errors. Any obstacles in the samples would prevent the accurate wavelength measurement. Alignment or positing of the samples is very important. Misalignment causes the wrong spectrograph. In particular, IR spectroscopy provides only the relative positions of a molecule's functional groups and will not determine the molecular weight of the samples.

Data analysis and management are one of the limitations. Collecting, managing, and deriving results from the data are very crucial for efficient chromatographic and spectroscopic techniques for determining the adulteration. Predictive

analytics to mine the data and build robust models are still inaccessible for laboratory data analysis. The unavailability of a systematic and structured collection of data and ontologies limits the data aggregation process.

Conclusion and Future Research Directions

During the last decade, substantial interest has been paid to virgin coconut oil quality and production methods owing to the recent crises and frauds in the food industry. This has seriously undermined the confidence of consumers. The official methods for detecting fraud are laborious and use harmful chemicals. The demand for rapid determination of authenticity and adulteration of CO and VCO using advanced analytical techniques is greater than ever; therefore, various spectroscopic, chromatographic, and other methods have been developed. Most of these techniques are non-destructive and give abundant information on both physical states and molecular structure, as well as a complete fingerprint of the product. Along with these techniques, chemometric tools have been applied for the targeted and untargeted analysis of the data to identify food fraud, and adulteration, or to trace its biological and geographical origins (authenticity). Among several chemometric tools, PCA was the most commonly used technique to discriminate against pure adulterated samples; PLS was widely used to quantify adulterant levels.

Table 3 An overview of commonly used chemometric tools

Chemometric method	Features	Advantages	Disadvantages	Application in VCO adulteration detection
Unsupervised pattern recognition				
PCA	PCA reduces the number of dimensions in the raw data set and generates new dimensions of data. The strongly correlated input variables are converted into uncorrelated values, known as principal components (PCs). The first PC can answer all the variability in the primary data set and the others can be neglected without significant loss of information	PCA gives an overview of a huge dataset. It can quickly determine if the dataset can be demonstrated by other linear classification methods	Class information is not taken into account. While calculating score and loading plot, non-linear combinations of variables are not considered. A scaling method is needed. In the case of lower variance, it could be misleading	(Amit et al. 2020a)
HCA	It is a clustering method organises the samples in groups, following a hierarchy. It is graphically represented as a dendrograms	HCA can give an outline of cluster of samples. Results interpretation is easy	Class information is not taken into account. It is highly sensitive for outliers. Its validation could be time consuming	(Mk et al. 2017)
Supervised pattern recognition				
LDA	LDA can classify data into two or more groups based on linear function. It is also called as discriminant function analysis	LDA is simple and quick. It is particularly suitable for low dimensional and linear data	In multi-classification task, it might lose its sensitivity. For higher dimensional data, it is not suitable. Non-linear information between variables and classes are not considered in LDA	(Amit et al. 2020b)
PLS-DA	PLS-DA, a discriminatory variant of PLS model and used for descriptive and predictive modelling	It is suitable for linear data. Mostly, it does not require variable selection method. It is useful in handling the collinearity among variables	The non-linear information among the peaks list is not considered. A scaling method is needed	(Lagurin et al. 2020)
SIMCA	SIMCA is based on PCA. It forms separate PCA models for each class or group. Classes were built based on the similarities (distances) between objects. It is represented as Coomans' plot and membership plot	Since it is a "soft" classification method, an object may be grouped into one, many, or no classes	Prior analysis by PCA is needed. It is highly sensitive to outliers	(Neves and Poppi 2020)
PLS-R	PLS-R is a multivariate statistical method. It builds a linear regression model by projecting the observed variables and the predicted variables to a space. It is utilised as a prognostic model	It has the ability to deal multi-collinearity in the data. Less restrictions in terms of statistics, like, sample size, level of measurements, and distribution of residuals	The predictive and response variables are defined without considering the variations which may exist within each dataset. Difficult to interpret solutions	(Amit et al. 2020a)

The conventional chromatographic techniques, which detect the marker compounds such as fatty acid methyl esters and triacylglycerols, for the identification of virgin coconut oil adulteration, are laborious, time consuming, and use harmful chemicals. Hence, rapid, precise, and environment-friendly vibrational spectroscopic techniques were developed. However, they generate a large amount of data that can only be managed by chemometrics, which is a tedious job. DSC was successfully employed to detect VCO adulteration, but it suffers from the disadvantage that it is not efficient in quantifying adulterants. Unfortunately, detecting the minute concentration of all possible adulterants using an ideal analytical technique is not feasible in reality. However, in the future, for rapid and effective screening of adulterated VCO at commercial and consumer levels, hand-held devices based upon digital imaging, vibrational spectroscopic techniques, and electronic tongue should be developed and applied.

The development of explicit, unique-purposed sensors and open-source software-based data collection and analysis systems will enable more sophisticated and tailor-made adulteration detection methods and devices. The combination of deep learning models and machine algorithms with artificial intelligence and cloud-based database management will reduce the burden of data analysis and derive the results. Image processing analysis of the oil samples and products prepared from the oil will be a quick adulteration detection and grading technique. Furthermore, the combination of electronic nose, E-tongue, and E-colour measurement with an electromyogram-based sensory system will help to detect and quantify adulteration in virgin coconut oils.

Acknowledgements The authors acknowledge the NIFTEM-T, Thanjavur, and ICAR-CPCRI, Kasaragod.

Declarations

Ethical Approval Not applicable.

Informed Consent Not applicable

Conflict of Interest Banu Priya declares no competing interests. Rashmitha declares no competing interests. Sai Preetham declares no competing interests. Chandrasekar declares no competing interests. Jagan Mohan declares no competing interests. Sinija declares no competing interests. Pandiselvam declares no competing interests.

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