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Evaluation of fresh palm oil adulteration with recycled cooking oil using GC-MS and ATR-FTIR spectroscopy: A review

Sook Ling Tan[1], Syazwan Hanani Meriam Suhaimy[2], Nur Azimah Abd Samad[3]*

[1]Department of Technology and Natural Resources, Faculty of Applied Sciences and Technology, Universiti Tun Hussein Onn Malaysia, Johor, Malaysia

[2]Department of Physics and Chemistry, Faculty of Applied Sciences and Technology, Universiti Tun Hussein Onn Malaysia, Johor, Malaysia

[3]Nanotechnology & Catalysis Research Centre (NANOCAT), Institute for Advanced Studies, University of Malaya, Kuala Lumpur, Malaysia

**Corresponding author: hananie@uthm.edu.my*

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Abstract: Palm oil (PO) is an edible vegetable oil that is extracted from the mesocarp of oil palm fruit (*Elaeis guineensis*), which is known to contain an almost equal proportion of saturated fatty acids (SFAs) and unsaturated fatty acids (USFAs). PO is used globally, because of its wide application as a frying medium. Extracted from the mesocarp of the oil palm fruit, PO needs to be processed to make it of edible quality. However, to meet growing global demand, it is often adulterated with recycled cooking oil (RCO), which is of inedible quality. As the methods of fresh palm olein (FPO) adulteration are sophisticated, it created an urgent need for commensurate analytical techniques with which to detect FPO adulteration. As such, chromatography and spectroscopy are commonly used to detect adulterations in edible oil. Therefore, this study evaluated the efficacy of utilising gas chromatography-mass spectrometry (GC-MS) and attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy to detect the adulteration of FPO with inedible RCO. Although previous studies attest to the efficacy of utilising GC-MS and ATR-FTIR spectroscopy in adulteration detection, both these techniques only provided specific qualitative and quantitative insights into the compounds present in oil samples. As such, further extensive studies on the application of a variety of adulteration detection methods are needed to provide regulatory authorities with information on the reliability of these modern adulteration detection methods.

Keywords: adulteration detection technique; fatty acid profile; Fourier transform infrared spectroscopy (FTIR); fresh palm olein; food security

Palm oil (PO) is derived from the mesocarp of the oil palm fruit (*Elaeis guineensis*). Upon collection, it is further refined physically and fractionated into a golden yellow-coloured end product that is safe for human consumption. PO contains almost equal portions of unsaturated fatty acids (USFAs) and saturated fatty acids (SFAs) (Mat Dian et al. 2017). Palmitic acid (C16 : 0) and oleic acid (C18 : 1) are SFAs and monounsaturated fatty acids (MUFAs) that, respectively, make up the majority of PO composition. Polyunsaturated fatty acids (PUFAs), specifically, linolenic acid (C18 : 3), make up about 11% of the total fatty acids present in PO.

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According to Tullis (2019), global demand for PO production has steadily increased over decades (from 12.5 million t in 1995 to 62.6 million t in 2015). This is due to the versatility as well as the highly available nature of PO. It is also more techno-economical than other oils and fats, especially soybean oil (Abdullah 2011). As Malaysia is one of the largest global producers of PO, PO is relatively popular in the domestic market as an edible frying oil. This, however, has enabled some unscrupulous traders and vendors to gain more profits by mixing a small portion of fresh palm cooking oil with recycled cooking oil (RCO). Over the past decade, the adulteration of fresh cooking oil with RCO was rampant in China before it prompted food safety concerns in other countries. This profit-driven fraudulent practice has also led to increased food safety concerns (specifically, fresh cooking oil adulteration) in Malaysia (Ramzy 2011; Lu et al. 2013; Legge 2014; Lu and Wu 2014).

RCO is oil that has been reprocessed from used or waste cooking oil (WCO) that has been heated multiple times. According to Lim et al. (2018), the long-term consumption of RCO promotes the formation of free radicals, which closely correlates with the high prevalence of chronic diseases, such as hypertension and cancer. As RCO has similar properties, such as colour density and reduced free fatty acids (FFAs), as that of fresh cooking oil, it is difficult to differentiate the two with the naked eye (Wannahari and Mad Nordin 2012). As households, restaurants, hotels, grocers, as well as other commercial and industrial food service establishments generate large amounts of WCO, it is recycled via the refining, bleaching and deodorising (RBD) process to recover the quality of the oil. However, RCO is intended for reuse in non-food applications, such as the manufacturing of biodiesel, soap etc., only. As such, it is normally sold at a relatively cheaper price than fresh cooking oil. This, unfortunately, has tempted some traders to adulterate fresh cooking oil with RCO before selling it as fresh cooking oil. Statistics show that 26.5% of scholarly articles on the incidence of food-related fraud between 1980 to 2012 (n = 1 648) were on cooking oils (Ebert 2013).

Traditional titrimetric methods, such as the quantitative chemical analysis of FFAs, total polar content (TPC), viscosity, saponification value, and specific gravity, as well as different oil quality parameters are commonly used to detect cooking oil adulteration. However, examining these properties using conventional detection methods is no longer practical due to limitations such as inaccuracy and insensitivity at lower adulteration concentrations. These methods of adulteration detection are also very time-consuming, use corrosive chemicals, as well as require intensive sample preparation (Azadmard-Damirchi and Tobarti 2015; Lim 2018; Yadav 2018). As the adulteration of fresh cooking oil has become more sophisticated, adulteration detection techniques that are as advanced and modern must be employed. Hong et al. (2017) suggest combining a two-step analytical method comprising of screening using Fourier transform infrared (FTIR) spectroscopy followed by a confirmation method, such as fatty acid profiling, using gas chromatography-mass spectrometry (GC-MS). Chromatography and fingerprinting FTIR spectroscopy have been widely used in adulteration detection due to economic advantages, better accuracy and sensitivity, minimal sample preparation, as well as good reproducibility. Previous studies have also attested to the efficacy of utilising GC-MS and FTIR (Zhang et al. 2014; Poiana et al. 2015; Hong et al. 2017; Lim et al. 2018).

Therefore, this study documents the application of GC-MS and attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy in detecting the adulteration of fresh PO with RCO as it is a food safety concern in Malaysia that requires immediate resolution.

ADULTERATION OF FRESH PALM COOKING OIL WITH RCO

As previously mentioned, the adulteration of fresh cooking oil with used cooking oil is commonplace in Asian countries. WCO is, generally, required to be reprocessed and purified via degumming, refinery, bleaching, and deodorisation (Zhao et al. 2015). This reprocessed WCO, or RCO, is intended for reuse in non-food applications, such as the production of biodiesel, soaps, and detergents. However, some irresponsible traders and vendors mix RCO into fresh palm olein (FPO) before repackaging it and selling it to consumers as fresh palm cooking oil (Lo 2017; Lim et al. 2018). This is a worrying issue as WCO is easily collected from restaurants, catering services, food stalls, and households.

In recent years, the adulteration of fresh cooking oil with RCO has quickly become one of the main food safety concerns in Malaysia. According to Lo (2017), used or WCO can be purchased at a relatively cheap price from households (between USD 0.12 per kg to USD 0.24 per kg). This low material cost as well as the simplicity of the WCO to RCO purification process has

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further facilitated cooking oil adulteration. As such, many unscrupulous traders and vendors have taken advantage of it to turn a quick profit. Cooking oil adulteration should also be taken seriously due to halal considerations; a chief concern among Malaysia's primarily Muslim consumers (Lim et al. 2018).

CHEMICAL CHANGES IN WCO

Due to repeated heating, the fatty acid composition (FAC) and quality of WCO is largely degraded. Although some of these properties can be recovered through recycling, there are still noticeable variations in the chemical composition of RCO in comparison with that of fresh cooking oil (Mannu et al. 2019).

FAC CHANGES IN WCO

When oils are subjected to high temperatures, such as during frying or cooking, the thermal stresses acting upon the oil break the *cis* double bonds (point of unsaturation) of USFAs, thereby, reducing the concentration of USFAs in the oil. Therefore, cooking oils with higher degrees of unsaturation are much more susceptible to autoxidation. The changes in FAC can be summarised as *i*) decreased concentrations of PUFAs and MUFAs, *ii*) increased concentrations of SFAs, and *iii*) increased concentrations of *trans* fatty acids (TFAs).

Giuffre et al. (2017) reported an identical decreasing trend of unsaturation in cooking oil with increased exposure time to thermal stress. Alireza et al. (2010) and Lim et al. (2018) also found similar fatty acid profile changes in heated oil. After prolonged frying time, the findings of both these experimental studies reported a significant decrease in PUFAs, such as linoleic (C18 : 2) and linolenic (C18 : 3) acids, as well as an increase in SFAs, such as palmitic (C16 : 0) and stearic (C18 : 0) acid. Abbas et al. (2016) found that longer exposure to heat resulted in greater quality degradation in corn oil as characterised by the weakening of *cis* double bonds. Poiana et al. (2015) reported similar findings when investigating extra virgin olive oil, soybean oil, and their blend of oil.

Apart from that, deep fat frying is also a recognised source of TFA production (Lim 2018). This is because *cis-trans* isomerism is induced at high frying temperatures leading to increased TFAs in the frying oil (Chen et al. 2014; Afaneh et al. 2017). This is corroborated by Giuffre et al. (2017) who noted that oil oxidation results in the degradation of USFAs into SFAs or the conversion of USFAs into TFAs. The accumulation of TFAs in the oil was explained by prolonged exposure to high temperatures (Martin et al. 2007; Chen et al. 2014).

FORMATION OF PRIMARY AND SECONDARY OXIDATION BY-PRODUCTS

Exposing cooking oil to high temperatures, such as frying, induces the production of hydroperoxides, the primary and unstable by-product of oxidation. This subsequently degrades into complex and more stable secondary by-products of oxidation, such as aldehyde, ketones, acids, and esters. At a high temperature, the thermal stress that acts upon the lipid causes an undesirable oxidation reaction which leads to decreased levels of unsaturation due to loss of the *cis* double bonds. The diminution of *cis* double bonds could be due to either the isomerisation of *cis-trans* isomers into *trans* isomers and/or the breakdown of double bonds that produce secondary oxidation by-products (Van de Voort et al. 1994). Poiana et al. (2015) found that the intensity of secondary oxidation by-products was at its highest at the end of the heating process due to the increased duration of heat exposure. This was characterised by the maximum absorbance of oil samples at the ATR-FTIR spectral band near 967 cm^{-1}, which alludes to the presence of secondary oxidation by-products.

REACTIVE MECHANISMS OF OIL QUALITY DEGRADATION

During the heating process, the oil is continuously subjected to a high temperature in the presence of atmospheric air and water. As such, reactions such as hydrolysis, oxidation, polymerisation, and *cis-trans* isomerisation occur resulting in the formation of undesirable volatile and non-volatile by-products of degradation (Choe and Min 2007; Nayak et al. 2015; Poiana et al. 2015; Boskou and Elmadfa 2016). The presence of primary by-products, such as FFAs and hydroperoxides, as well as secondary oxidation products further degrade the quality of the oil in terms of nutritional value as well as sensory and functional quality. As a whole, deep fat frying deteriorates the quality of oil (Nayak et al. 2015). The degradation of oil quality upon frying is the reason why WCO should be discarded eventually rather than reuse for human consumption. Lim (2018) points out that these degradation compounds may be present in RCO as it is reprocessed from WCO, which is normally used for frying and cooking.

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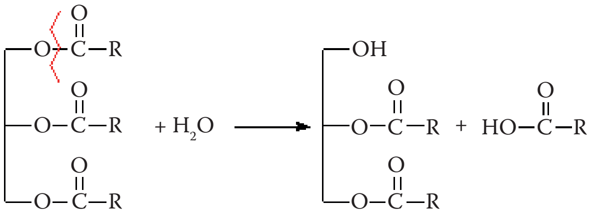


Figure 1. The formation of fatty acids and diacylglycerols

Hydrolysis. As it is often used as a frying medium, PO is exposed to high temperatures in the presence of air and moisture. As the moisture found in fried food items comes into contact with the frying oil, it induces hydrolysis. During the heat and mass transfer that occurs during the frying process, oil is absorbed into the fried food while moisture is removed (Budzaki and Seruga 2005; Oke et al. 2017). The presence of moisture in the frying oil catalyses the hydrolysis of the oil breaking it down into FFAs, glycerol, monoglyceride, and diglyceride (Figure 1) (Thapar 2019).

Chung et al. (2004) found a direct correlation between the amount of FFAs and the number of frying cycles, where the amount of FFAs increased with the number of frying cycles. The extent of hydrolysis depends on factors such as oil temperature, interface area between the oil and the aqueous phase, as well as the amount of water and steam. The presence of FFA and other oxidised compounds cause the oil to have an off-flavour which makes it less acceptable for deep fat frying. The presence of FFA, glycerol, mono-, and diglycerides further enhance the hydrolysis of the oil resulting in a continuous degradation in oil quality with every subsequent frying cycle (Frega et al. 1999; Nayak et al. 2015).

Oxidation. Lipid oxidation is the major cause of quality deterioration in oil that has been used for deep frying. The three main types of oxidations that occur in deep-frying oil are lipid autoxidation, thermal oxidation, and photooxidation. Autoxidation occurs when oil comes into direct contact with oxygen at an atmospheric temperature resulting in rancidity (Nayak et al. 2015). Thermal oxidation takes place at temperatures exceeding 180 °C, which is the typical frying temperature. The chemical mechanism of thermal oxidation is principally identical to that of autoxidation, which involves a free radical reaction (Figure 2). Nevertheless, the rate of thermal oxidation is faster than that of autoxidation (Choe and Min 2007). This mechanism involves three main steps: initiation, propagation, and termination.

During initiation, when lipid molecules (RH) are exposed to initiators such as heat, light, and metal catalysts, the hydrogen atoms of the double bonds are abstracted to form alkyl free radicals (R^-) (Nayak et al. 2015; Madhujith and Sivakanthan 2019). In the subsequent propagation step, these alkyl free radicals react with oxygen to form peroxy radicals (ROO^-). These peroxy radicals then abstract hydrogen from other lipid molecules (RH) and react to form the primary oxidation by-product, hydroperoxide (ROOH), as well as another alkyl radi-

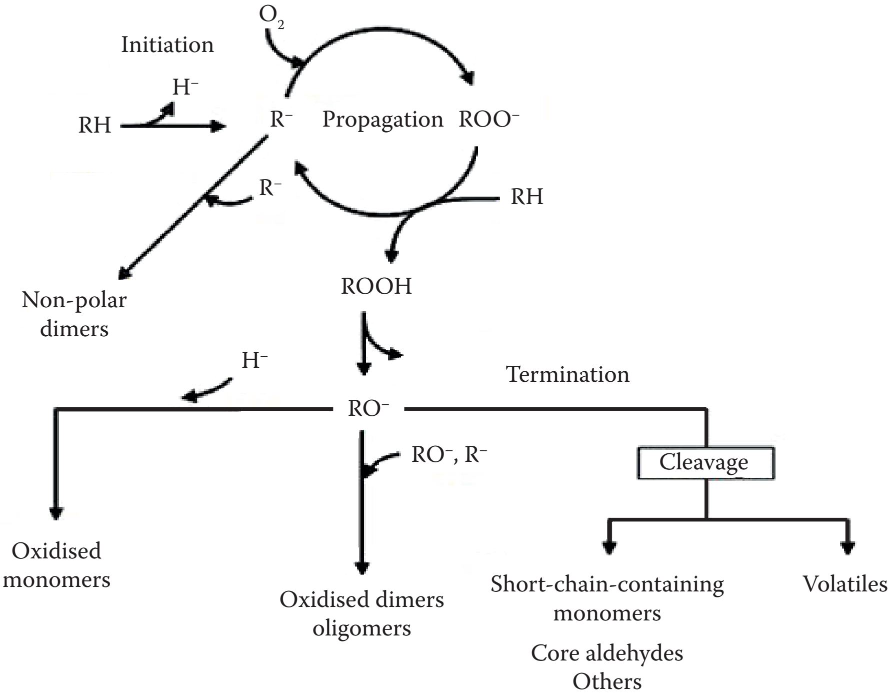


Figure 2. Simplified scheme of thermal oxidation (Dobarganes 2009)

cal (R^-) (Nayak et al. 2015). However, as hydroperoxides are very unstable, they break down into short-chain compounds via the cleavage of oxygen-oxygen (O-O), carbon-carbon (C-C), and carbon-oxygen (C-O) bonds around the peroxide group (Choe and Min 2007; Nayak et al. 2015). Meanwhile, the radicals further catalyse the oxidation reaction (Choe and Min 2007; Nayak et al. 2015; Madhujith and Sivakanthan 2019).

As these non-volatile and odourless hydroperoxides are highly unstable, they further break down into more complex and stable secondary oxidation by-products called carbonyl compounds, such as aldehyde, ketones, alcohols, esters, and acids. Hydroperoxides begin to decompose into alkoxy radicals (RO^-) and hydroxyl radicals (OH^-) as soon as they are formed (Choe and Min 2007; Madhujith and Sivakanthan 2019). The alkoxy radicals that are formed either react with other alkoxy radicals or decompose to form non-radical products. The formation of non-radical final products marks the end of the oxidation process, the termination step.

Polymerisation. Polymers are high conjugated dienes (CDs) that form a brown, resin-like residue along the sides of fryers when the oil and metal come into contact with oxygen in the air (Choe and Min 2007). The polymers formed during deep-fat frying are rich in oxygen. These oxidised polymers, the end products of thermal polymerisation during frying, were known to accelerate the rate of oil oxidation (Yoon et al. 1988). The presence of these polymers not only further deteriorates oil quality, increases viscosity, and reduces heat transfer efficiency but causes foaming and discolours the food as well as exponentially increases the oil uptake of the food which is bad for health (Choe and Min 2007).

Choe and Min (2007) noted that the major by-products of decomposition present in frying oil are non--volatile polar compounds as well as triacylglycerol dimers and polymers. Dimers and polymers are large compounds with high molecular weights, ranging between 692 to 1 600 Daltons (Da), that possess hydroperoxyl, epoxy, hydroxy, and carbonyl groups as well as -C-O-C- and -C-O-O-C- linkages (Choe and Min 2007). In deep-fat frying, dimerization and polymerisation are radical reactions. Khor et al. (2019) reported that polymerised triacylglycerols are associated with a high prevalence of chronic diseases, such as atherosclerosis, liver damage, and diabetes, as well as the proliferation of intestinal tumours. The formation of dimers and polymers in frying oil depends on factors such as oil type, frying temperature, and the number of frying cycles (Choe and Min 2007). Takeoka et al. (1997) reported a direct correlation between polymer accumulation and the number of frying cycles and temperature. A steady increase in the number of polymers was observed as the number of frying cycles and temperature increased. The fatty acid profile of a frying oil was found to affect the formation of polymers, where oils high in linoleic acid (exceeding 20%) produced a significant quantity of polymers (Takeoka et al. 1997). Therefore, the more unsaturated the oil, the more likely it is to produce polymers.

***Cis-trans* isomerisation.** TFAs are a group of USFAs that comprise of one or more isolated, non-conjugated double bonds in a trans geometric configuration (Chen et al. 2014). Multiple studies have found a direct correlation between TFAs and cardiovascular diseases, breast cancer, diabetes, shorter gestation during pregnancy, colon cancer, allergies and so on (Dhaka et al. 2011). This illustrates the detrimental health effects of TFAs. As such, consumers should avoid it. *Cis-trans* isomerism occurs during frying at high temperatures and increases the amount of TFAs in oil containing USFAs (Chen et al. 2014; Afaneh et al. 2017). However, the formation of *trans* compounds depends on frying time and temperature. Martin et al. (2007) suggested that the formation of *trans* isomers increases with frying time. Meanwhile, Chen et al. (2014) found significant *trans* isomers accumulation at frying temperatures above 250 °C. This could be because a particular amount of energy is required to convert the double bonds in fatty acids from a *cis* to *trans* configuration (Chen et al. 2014).

Apart from frying temperature and time, other factors such as oil type, FAC, and the presence of antioxidants could also influence TFA accumulation. Similarly, the more unsaturated an oil, the more susceptible it is to *cis-trans* isomerisation during deep-fat frying. Therefore, an excellent frying medium should have a low level of PUFAs, such as linoleic (C18 : 2) and linolenic (C18 : 3) acids, high levels of oleic acid (C18 : 1), and moderate amounts of SFAs (Latha and Nasirullah 2014). This suggests that oleic-rich oils, such as palm olein, is preferable for frying due to their excellent oxidative stability.

MODERN ANALYTICAL TECHNIQUES OF ADULTERATION DETECTION

As the adulteration of fresh cooking oil has become more sophisticated, chemical titrimetric methods that characterise changes in the physical, chemical, and physicochemical properties of oils are outdated and no longer practical. Therefore, modern analytical approaches such as fingerprinting FTIR spectroscopy and chromatography are widely implemented these days for rapid and effective adulteration detection in fresh cooking oil.

FAC analysis using GC-MS. Chromatographic methods are more commonly used to determine FPO adulteration (Che Man et al. 2005; Lim et al. 2018). Other than that, GC is also popularly used to detect adulteration in different oils, such as extra virgin olive oil, camellia seed oil, and canola oil (Yang et al. 2013; Li et al. 2016; Chen et al. 2018). As such, the efficacy of GC in adulteration detection is proven.

GC can be coupled with numerous types of detectors, such as a flame ionisation detector (GC-FID), mass spectrometry (GC-MS), or ion mobility spectrometry (GC-IMS). This study, however, only investigated the efficacy of GC-MS in detecting the adulteration of FPO with RCO. According to Lim (2018), GC-MS is the more preferable method of adulteration detection as it is more accurate. It is also a promising high-end detection tool that is applicable in many fields. GC methods are highly sensitive and challenging, as professional experiments are required to operate the device. Sample pre-treatment is time-consuming and may involve some corrosive chemical reagents. Acid- and base-catalysed transmethylation processes are commonly cited in the literature for conversion of fatty acids into fatty acid methyl esters (FAME). Lim et al. (2018) and Zhang et al. (2014) prepared FAME using methanolic sulphuric acid (acid catalyst) and potassium hydroxide methanol solution (base catalyst) separately, which were reported to have good derivatisation efficiency.

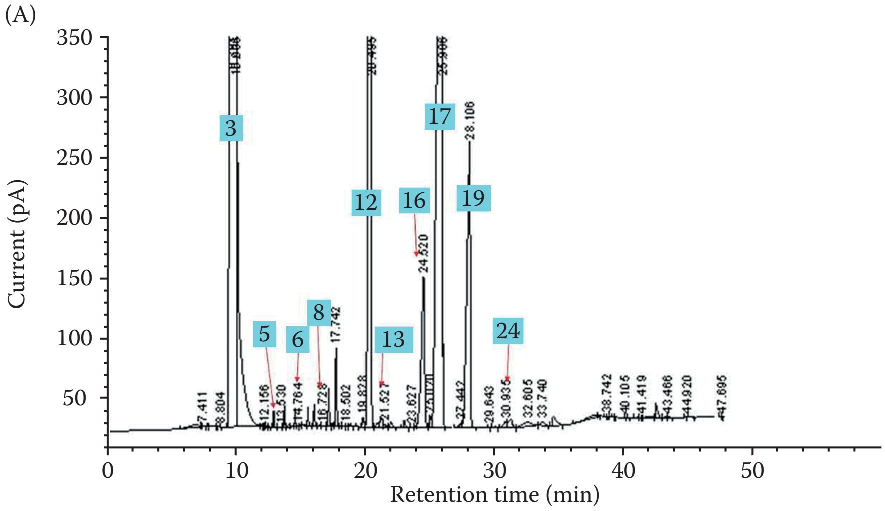
FAC is a useful purity indicator with which to determine oil quality. The composition of FAME can be characterised by GC. Additionally, the percentage of PUFAs such as linoleic and linolenic acid could be utilised as a parameter for detecting FPO adulteration with RCO. Lim et al. (2018) reported the experimental observation of a consistent decreasing trend of PUFAs with an increasing adulteration level of RCO. This further supported that PUFAs could be a potentially useful indicator for the detection of adulteration in FPO. Importantly, PUFAs could be a potential indicator for the detection of adulteration in other edible oils such as olive oil, as reported by Salah and Nofal (2020). Previous studies have shown that FAC analysis is a promising analytical parameter with which to detect adulterants in edible oil, even at low levels of adulteration. This is because FAC analysis determines the fatty acid content of oil then quantifies it as a percentage of the total fatty acids in the oil (Zhang et al. 2014; Azadmard-Damirchi and Torbati 2015; Lim et al. 2018). For GC methods, multivariate chemometric methods such as principal component analysis (PCA), partial least-squares-discriminant analysis (PLS-DA) are often used in combination to classify oil types and recognise the chromatographic patterns. As GC-MS evaluates an oil sample both qualitatively and quantitatively, it can qualitatively characterise the FAC of the oil sample as the peaks obtained in the chromatographic analysis will be identified by comparing it with the retention times of an authentic oil to determine the presence of specific fatty acids in the sample (Bahadi et al. 2016; Bazina and He 2018; Medeiros 2018). This quantitated FAC can then be compared with the Codex standard to determine the extent of adulteration.

The method of GC-MS and GC-FID was adopted by Zhang et al. (2014) and Lim et al. (2018) respectively, to detect adulteration in olive oil and palm olein through FAC and its content. Zhang et al. (2014) and Lim et al. (2018) combined with chemometrics of random forests (RF) and discriminant analysis (DA) respectively, to classify the oil samples and identify adulterants. Lim et al. (2018) compared the FAC of adulterant (RCO) with FPO through fatty acid profiles. Patterns such as peak and area of the peak were observed and compared with standard, to identify the changes in FACs with an increasing adulteration level. Through FAC analysis, Lim et al. (2018) compared FPO, RCO and adulterated oil samples with different adulterant levels among each other and with the Codex standard, to detect the adulteration.

With regard to FPO adulteration with RCO, Lim et al. (2018) suggested evaluating a combination of fatty acids as an indicator as noticeable changes were observed in FPO, RCO, and adulterated oil. The study also reported the appearance of short-chain fatty acids (SCFA) and TFA due to the thermal oxidation of the WCO and RCO, which is reprocessed from WCO. A significant increase in MUFA, as well as an insignificant yet consistent decrease in PUFA, was also observed. Therefore, FAC analysis using GC-MS was potentially useful and effective at detecting cooking oil adulteration by evaluating a combination of fatty acids.

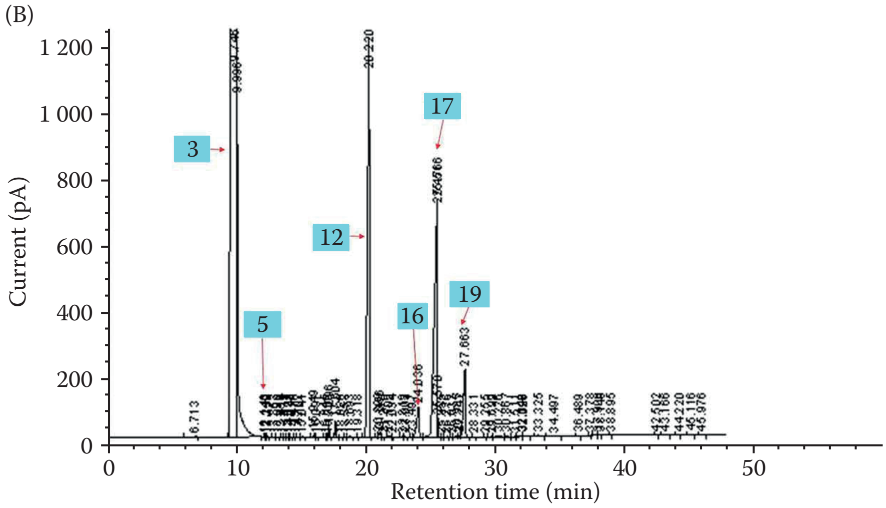
Chromatographic analysis of FPO and RCO. Abidin et al. (2013), as well as Lim et al. (2018), characterised the properties of used cooking oil and RCO, respectively. As previously mentioned, RCO is retrieved and further processed from used or WCO. Although RCO has undergone a certain degree of modification, both WCO and RCO may share similar oil properties as they are both from the same source. Figure 3 shows

(A)



Peak number	Fatty acids	Number of carbon
3	caprylic	C8 : 0
5	undecanoic	C11 : 0
6	lauric	C12 : 0
8	myristic	C14 : 0
12	palmitic	C16 : 0
13	palmitoleic	C16 : 1
16	stearic	C18 : 0
17	oleic	C18 : 1 n-9 *cis*
19	linoleic	C18 : 2 n-6 *cis*
24	*cis*-11-eicosenoic	C20 : 1 n-9

(B)



Peak number	Fatty acids	Number of carbon
3	caprylic	C8 : 0
5	undecanoic	C11 : 0
12	palmitic	C16 : 0
16	stearic	C18 : 0
17	oleic	C18 : 1 n-9 *cis*
19	linoleic	C18 : 2 n-6 *cis*

Figure 3. Gas chromatogram of fatty acid profile analyses of (A) fresh palm olein (FPO) and (B) recycled cooking oil (RCO) (Lim et al. 2018)

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a comparison of FAC between FPO and RCO. Several noticeable, albeit minor, changes were detected in the FAC of RCO in comparison with FPO. The significant peaks that were observed in the FAC of RCO were also detected in that of FPO. However, Lim et al. (2018) reported that the trace amounts of certain fatty acids present in RCO [caprylic acid (C8 : 0), capric acid (C10 : 0), undecanoic acid (C11 : 0), elaidic acid (*trans* C18 : 1), and eicosapentaenoic acid (C20 : 5)] were not detected in FPO. The differences in fatty acid profiles, as evidenced by the presence and concentrations of specific fatty acids, suggested that analysing combinations of fatty acids could potentially be used as an indicator of adulteration.

FAC evaluation is critical to observe the trend of some interested fatty acids such as linoleic acid and linolenic acid in FPO, RCO and adulterated oil with different adulteration levels (Lim et al. 2018). The formation of TFA in RCO may be the consequence of *trans*-isomerisation from frying and deodorising at a high temperature. In exposure to high heat during the frying process, the lipid degradation process takes place to break down triacylglycerols into FFAs. The breakdown of double bonds due to heat stress subsequently caused a decrease in PUFAs concentration.

Figure 4 depicts the typical chromatogram of used or WCO (Abidin et al. 2013). As seen, the five predominant fatty acid components present in used cooking oil are palmitic acid (C16 : 0), stearic acid (C18 : 0), oleic acid (C18 : 1), linoleic acid (C18 : 2), and linolenic acid (C18 : 3). The similar FACs found in the used cooking oil, FPO, and RCO indicated that these oils were from the same source, where the minor difference in oil properties may be due to frying and refining.

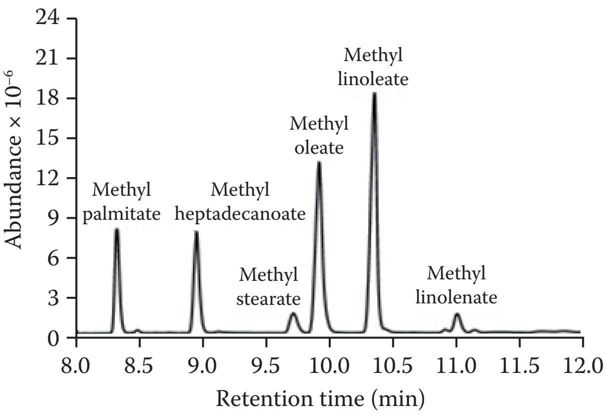

Figure 4. Typical gas chromatogram of derivatised used cooking oil (Abidin et al. 2013)

Evaluation of functional groups using ATR-FTIR spectroscopy. FTIR spectroscopy is an ideal method of characterising the chemical structural properties of analyte compounds and evaluating their functional groups for a chemical characterisation purpose. FTIR is a simple analytical technique that provides rapid and accurate results. As such, multiple previous studies have comprehensively used FTIR spectroscopy to determine the authenticity of various types of oil, such as extra virgin olive oil, FPO, virgin coconut oil, and cold-pressed sesame oil (Poiana et al. 2015; Ozulku et al. 2017; Lim et al. 2018; Amit et al. 2019).

FTIR spectroscopy provides both qualitative and quantitative insight into the functional group structure and composition of chemical compounds present in food samples (Mashodi et al. 2020). It also provides the qualitative data of an oil sample by identifying the organic groups present via the vibration of molecular bonds due to the presence of certain functional groups in the infrared spectrum at characteristic wavenumber (Mashodi et al. 2020). As each type of bond absorbs radiation and vibrates at a characteristic wavenumber, fingerprinting FTIR spectroscopy is capable of identi-

Table 1. Aberrations in FTIR bands to be investigated in detecting FPO adulteration with RCO

Wavenumber (cm^{-1})	Functional group	Mode of vibration	Indication
3 529	O-H	stretching	presence of secondary oxidation product or alcohol associated with thermal oxidation
3 442	O-H	stretching	formation of primary oxidation product, hydroperoxide
1 655	-C=C- (*cis*)	stretching	change in *cis* double bond or unsaturation level
988	-HC=CH- (*trans, trans*)	bending	presence of conjugated *trans-trans* double bond or *trans* isomers
964 (FPO), 968 (RCO)	-HC=CH= (*trans*)	bending out of plane	presence of *trans* double bond
908 (FPO), 904 (RCO)	-HC=CH- (*cis*)	bending out of plane	presence of *cis* carbon-carbon double bond

FTIR – Fourier transform infrared spectroscopy; RCO – recycled cooking oil; FPO – fresh palm olein fying the functional groups of a molecule. Meanwhile, the quantitative data is based on the intensities of the bands in the infrared spectrum, where Lopes et al. (2018) reported that the concentration of the functional groups present is directly proportional to its absorbance in the spectrum in accordance with Beer's Law.

Nowadays, FTIR spectroscopic technique has been widely used in detecting edible oils adulteration for its advantages: rapid, easy and minimal sample preparation required. Poiana et al. (2015) and Lim et al. (2018) had employed ATR-FTIR to differentiate pure and adulterated oils. Lim et al. (2018) reported the FTIR spectra were measured from 4 000 cm^{-1} to 650 cm^{-1} at a controlled ambient condition, with a setup of 32 scans and resolution of 1 cm^{-1}, to investigate adulteration in FPO.

Advanced sampling techniques of acquiring FTIR spectra have resulted in the development of new and helpful accessories that simplify the sample preparation procedure. However, despite a variety of techniques and accessories, ATR is still commonly utilised for liquid samples (Shimadzu Corporation 2020). When using ATR-FTIR spectroscopy, a liquid oil sample can be dropped on the surface of the ATR crystal, which has a high refractive index such as that of a diamond crystal, to avoid unnecessary disturbance. Therefore, the surface of the ATR crystal will allow infrared light to pass through it and interact with the sample directly, resulting in FTIR spectra. Furthermore, the difference in the refractive indices of the ATR crystal and the sample will result in total internal reflectance.

When utilising ATR-FTIR to detect the adulteration of FPO with RCO, the infrared (IR) spectra of the oil sample was usually recorded at the 4 000 cm^{-1} to 650 cm^{-1} wavenumber regions. This is, therefore, a useful indicator of adulterant detection in edible oil (Rohman and Che Man 2010; Liang et al. 2012; Lim et al. 2018). Poiana et al. (2015) and Lim et al. (2018) found that the spectra of both FPO and RCO were similar in terms of shape and the position of characteristic bands as both oils shared the same origin. However, spectral differences were still observed at the wavenumber regions as a consequence of adulteration with low-quality oil (Table 1). Different oils have different FACs and functional groups (Salah and Nofal 2020). Adulteration of FPO with RCO will degrade the oil quality, as RCO has undergone a harsh frying procedure in the commercial frying industry. As a consequence of fat degradation, oxidation products were formed and oil composition was altered. Therefore, FTIR spectral analysis is a potentially reliable tool with which to detect and quantify functional groups in FPO that has been adulterated with RCO (Lim et al. 2018). The small aberrations in FTIR spectral bands and absorbance intensities revealed the presence of adulterant in FPO samples.

FTIR spectral analysis of FPO and RCO. Figure 5A shows the FTIR spectra of FPO and RCO at the 4 000 cm^{-1} to 650 cm^{-1} mid-infrared wavenumber region (Lim et al. 2018). Both FPO and RCO demonstrated similar spectral features, in terms of shape and

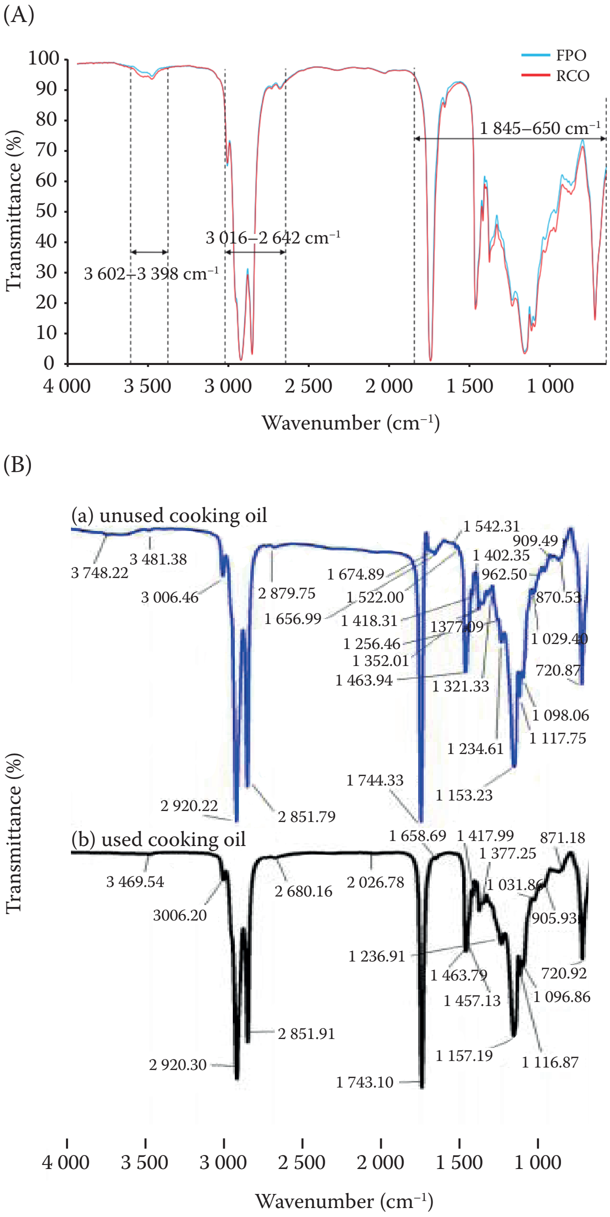

Figure 5. Comparison of FTIR spectra of (A) FPO and RCO at mid-infrared region of 4 000–650 cm^{-1} (Lim et al. 2018) and (B) fresh cooking oil and used cooking oil (Ullah et al. 2014)

FTIR – Fourier transform infrared spectroscopy; RCO – recycled cooking oil; FPO – fresh palm olein position of the characteristic bands, as they share the same origin. The striking similarity between the FTIR spectra of both FPO and RCO was also due to the presence of triglycerides, the principal component of both the oils (Ullah et al. 2014). Used cooking oil, which has not been further refined into RCO, has a similar spectrum as fresh cooking oil, as observed by past studies (Ullah et al. 2014; Abdul Wahab et al. 2015). This suggests that the infrared spectra of these oils are similar when observed with the naked eye because of their similar chemical composition in terms of fatty acid.

Nevertheless, due to their distinctive triglyceride compositions, slight differences are observable between FPO, RCO, and used cooking oil in terms of the exact position of characteristic bands and absorbance intensities. Lim et al. (2018) reported that, with respect to absorbance intensity, appreciable variations were detected between FPO and RCO at certain wavenumber ranges of the spectral region, specifically 3 602 cm^{-1} to 3 398 cm^{-1}, 3 016 cm^{-1} to 2 642 cm^{-1}, and 1 845 cm^{-1} to 650 cm^{-1}. This was corroborated by Ullah et al. (2014) where different oils had slight differences both in the exact position and absorbance of the bands due to distinctive triglyceride compositions. The spectral alteration of these oils is due to the degradation of their properties from prolonged exposure to high heat during frying.

ADVANTAGES AND LIMITATIONS

As edible oils that are meant for human consumption are frequently counterfeited using sophisticated techniques, modern analytical techniques, such as chromatography and spectroscopy, have been employed to detect edible oil adulteration, thereby, replacing traditional chemical titrimetric methods (Hong et al. 2017). Although these modern analytical techniques are more sensitive, accurate, and efficient, there are not without their limitations (Table 2).

Comparison of traditional and modern analytical techniques. A number of parameters, such as FFA content, peroxide value, iodine value, saponification value, acid value, and viscosity, can be used as indicators of oil quality. Determination of chemical indices such as FFA, acid value and peroxide value are commonly cited in the literature to evaluate the changes in oil properties. Maskan and Bagci (2003) and Nayak et al. (2015) suggested that increment in FFA concentration indicated oil quality degradation. RCO is reprocessed from used frying oil, which is characterised by high FFA content when compared with fresh cooking oil. In the course of the intensive frying process, frying oil is exposed to high heat and cause the TAG degradation into FFA. Hence, determination of chemical indices such as FFA analysis is proved to be feasible in detecting adulteration incidence, as adulterated oil with RCO was expected to exhibit relatively higher FFA content when compared with FPO.

However, these conventional titrimetric methods do not lack the sensitivity to detect lower concentrations of adulterants but they are time-consuming as well. As adulteration methods have become more sophisticated, the development and application of modern analytical techniques have helped overcome shortcomings as well as improve the efficacy of cooking oil adulteration detection. Table 3 provides a comparison of the traditional and modern adulteration detection techniques used for edible oils.

Table 2. Advantages and limitations of GC-MS and ATR-FTIR spectroscopy

Analytical techniques	Advantages	Limitations
GC-MS	– provide better resolution and separation for peaks – lower limit of detection (lod) – provide rich information of qualitative and quantitative analysis of sample – robustness – offer greater sensitivity and specificity – high reproducibility	– massive and lengthy sample preparation (hydrolysis and derivatisation of sample) – destructive method – time-consuming for sample analysis – incapable for compounds that are non-volatile, polar or thermally labile – identified compounds have to be in mass spectral library/database for identification purpose
ATR-FTIR	– simplicity, rapid, economic, good reproducibility – minimal/almost no sample preparation required – non-destructive method – provide both qualitative and quantitative information	– ATR spectra need further processing in order to obtain significance on a molecular level – selection of ATR crystal will influence the resulting spectra

GC-MS – gas chromatography-mass spectrometry; ATR-FTIR – attenuated total reflection-Fourier transform infrared

Table 3. Comparison of conventional and modern adulteration techniques in edible oil adulteration

Method/ indicator	Cooking oil studied	Adulterant	Advantage	Limitation	References
Conventional chemical technique					
FFA	palm olein	RCO	–	– inaccurate measurement as the identity of adulterant could be easily masked by adding adsorbent and a small portion of fresh cooking oil	(Lim 2018)
TPC	palm olein	RCO	–	– an indicator used to measure the degree of an overall degradation of oil samples, unable to distinguish fresh cooking oil and those adulterated with RCO	(Lim 2018)
Saponification value	sesame oil	sunflower oil	– adulteration can be easily identified at a concentration above 10%	– insensitive to lower adulteration range (5–10%), adulteration at low concentration is not easily identifiable – maximal sample preparation step and chemical usage	(Shotorbani et al. 2018) (Yadav 2018)
Iodine value	mixed edible oil (soybean oil, olive oil, peanut oil, blend oil)	–	– measure the degree of unsaturation, which is important to assess the quality of oil and authentication test	– ineffective since it uses highly toxic chemicals that are environmentally unfriendly – method is complex and time-consuming	(Yan et al. 2018) (Yadav 2018)
Modern analytical technique – Chromatographic					
GC-FID coupled with chemometrics	palm olein	RCO	– fatty acid profile of oil sample is identifiable – previous studies showed the method perform well in detecting adulterated oil sample	– less accurate compared to that coupled with MS detector	(Lim 2018)
GC-MS	camellia seed oil	soybean oil	– simple, accurate and rapid – LOD of adulteration as low as 5% – provide better resolution and separation for peaks compared to GC-FID	– fatty acids need to be evaluated in combination as valid indicators to detect adulteration; otherwise can get an incorrect authentication result	(Xie et al. 2012) (Lim 2018)
Modern analytical technique – Spectroscopic					
ATR-FTIR	extra virgin olive oil	soybean oil	– fast, low cost per analysis – minimal sample preparation compared to KBr-FTIR – able to study the structural changes induced in oil by adulteration; promising mean to differentiate pure and adulterated oil	–	(Poiana et al. 2015)

FFA – free fatty acid; TPC – total polar content; RCO – recycled cooking oil; GC– gas chromatography; MS – mass spectrometry; FID – flame ionisation detector; LOD – limit of detection; ATR-FTIR – attenuated total reflection-Fourier transform infrared; KBr – potassium bromide

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CONCLUSION

The adulteration of FPO with RCO has quickly become a major food safety concern in Malaysia. However, studies on this specific topic are scarce. A recent study on detecting and quantifying the adulteration of FPO with RCO proved the practicality and efficiency of advanced analytical detection methods in adulteration detection. Modern analytical techniques, such as GC-MS and ATR-FTIR spectroscopy, have proven to be effective at adulteration detection with rapid, sensitive, and accurate results in comparison to traditional chemical titrimetric methods. Future studies on FPO adulteration with RCO should focus on a variety of adulteration detection techniques in order to furnish regulatory authorities with information on the reliability of modern instruments that may aid the detection of adulterants in edible oils.

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