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ANALYTICAL TECHNIQUES FOR DETECTION OF ADULTERATION IN CRUDE PALM OIL

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By

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Chair : Professor Dr. Lai Oi Ming, PhD
Faculty : Biotechnology and Biomolecular Sciences

In recent years, the demand and consumption for Crude Palm Oil (CPO) has gradually increased in world market. However, production to fulfill the rising demand is rather slow. The gap between demand and production thus lead to potential frauds or adulteration of the CPO. Therefore, we are investigating the possibility of using analytical techniques such as gas chromatograph (GC), high performance liquid chromatograph (HPLC), differential scanning calorimetric (DSC) and Fourier transform infrared (FTIR) spectroscopy to detect CPO adulteration with sludge oil (SO) and used vegetable oil (UVO/UVOA). Investigations conducted with individual fatty acids and triacylglycerol by GC and HPLC analysis, respectively showed that it was very difficult to distinguish between genuine CPO from adulterated CPO. However, combination of fatty acids and triacylglycerols with multivariate statistical analysis produced more promising results. With these techniques, contaminated CPO with adulterants between 2-5% was easily detected by principle component analysis (PCA) and cluster analysis (Dendrogram). DSC thermograms showed that detection of adulteration was difficult to be determined by using heating thermogramd due to the polymorphism of oils and fats. However, the profile of DSC cooling thermograms showed changes in peak intensity as the amount of adulterant concentrations varied in CPO. This provided a positive indication that detection of adulterants in CPO could be determined from the changes in peak intensity. In parallel, application of multivariate and regression analysis were able to identify adulterant contents as low as 2% accurately in CPO. FTIR spectroscopy was the last technique applied to detect SO and UVO/UVOA adulteration. FTIR techniques seems to be the least promising technique to detect adulteration of CPO. However adulteration with used vegetable oil containing animal fat (UVOA) was clearly highlighted and was able to be discriminated by cluster analysis Dendrogram at level of 2 % using FTIR spectra. Nevertheless, further study is required to improve the level of detection as well as accuracy for discriminating genuine CPO from the adulterated CPO.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

TEKNIK ANALISA UNTUK MENGESAN PENGADUKAN DALAM MINYAK SAWIT MENTAH

Oleh

ANAND KUMAR INTHIRAM
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Semenjak kebelakangan ini, permintaan dan penggunaan untuk minyak sawit mentah (CPO) telah beransur-ansur meningkat di pasaran dunia . Walau bagaimanapun, pengeluaran CPO bagi memenuhi permintaan menjadi suatu cabaran . Jurang di antara permintaan dan pengeluaran CPO membawa kepada penipuan atau pencampuran minyak murah yang lain di dalam CPO. Oleh itu, potensi menggunakan teknik analisa seperti kromatografi gas (GC) , kromatografi cecair prestasi tinggi (HPLC) , permeteran kalori pengimbasan kebezaan (DSC) dan Spektoskopi infra-merah (FTIR) untuk mengenalpasti pencampuran CPO dengan minyak enapcemar (SO) dan minyak sayuran terguna (UVO/UVOA) disiasat dalam kajian ini. Kajian yang dijalankan dengan menentukan komposisi asid lemak dan kandungan trigliserida dengan analisis GC dan HPLC, masing-masing menunjukkan bahawa ia adalah amat sukar untuk membezakan CPO tulen daripada CPO yang diaduk. Walau bagaimanapun, gabungan penentuan asid lemak dan kandungan trigliserida dengan analisis statistik multivariat menghasilkan keputusan yang lebih merangsangkan . Dengan aplikasi ini , CPO diaduk dengan bahan aduk di antara 2 hingga 5% boleh dikesan melalui analisis komponen prinsipal (PCA) dan analisis kelompok (dendrogram). Termogram pencairan DSC menunjukkan bahawa pengesanan pencemaran adalah sukar untuk dikenal pasti disebabkan oleh sifat polimorfik minyak dan lemak. Walau bagaimanapun , profil termogram penyejukan DSC menunjukkan perubahan pada intensiti puncak apabila jumlah kepekatan minyak aduk seperti SO dan UVO/UVOA meningkat di dalam CPO. Ini memberikan satu petunjuk yang positif bahawa pengesanan bahan asing dalam CPO boleh ditentukan melalui perubahan intensiti puncak. Pada masa yang sama , penggunaan analisis multivariat dan regresi dapat mengenal pasti kandungan bahan aduk serendah 2 % dalam CPO. Spektroskopi FTIR adalah teknik terakhir yang digunakan untuk mengesan SO dan UVO / UVOA. Teknik FTIR yang digunakan tidak berkesan untuk mengenai pasti bahan adukan dalam CPO. Walau bagaimanapun pencemaran dengan minyak sayur-sayuran yang digunakan yang mengandungi lemak haiwan (UVOA) dapat diskriminasi melalui analisis kelompok , dendrogram di peringkat 2%. Walau bagaimanapun , kajian lanjut diperlukan untuk meningkatkan tahap pengesanan dan ketepatan pengesan CPO tulen daripada yang diaduk.

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APPROVAL SHEETS (Replace with printed copy from UPM)

I certify that a Thesis Examination Committee has met on 13th March 2015 to conduct the final examination of ANAND KUMAR INTHIRAM on his thesis entitled "DETECTION OF CRUDE PALM OIL ADULTERATION BY CHEMICAL PROPERTIES USING CHROMATOGRAHIC, THERMAL AND SPECTRAL TECHNIQUES." in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

CPO	Crude Palm Oil
PCA	Principle Component Analysis
SIMCA	Standard in Multivariate Data Analysis
PLS	Partial Least Square
GC	Gas Chromatograph
HPLC	High Performance Liquid Chromatograph
FT-IR	Fourier Transform Infrared
NMR	Nuclear Magnetic Resonance
NIR	Near Infrared Spectroscopy
DNA	Deoxyribonucleic Acid
FAME	Fatty Acid Methyl Esters
DSC	Differential Scanning Calorimetry
PCR	Polymerase chain reaction
SNP	Single nucleotide polymorphism
UVO	Used Vegetable Oil
UVOA	Used Vegetable oil Containing Animal Fats
SO	Sludge Oil
CI	Confident Interval
Df	Degree of Freedom

CHAPTER 1

INTRODUCTION

In the recent years, a great interest has been devoted to the use of vegetable fats in human diet, especially regarding olive oil and other vegetable edible oil. Since these oils have a vast implication for human health due to their nutritional properties. Most olive oil and some vegetable oils are rich in monounsaturated fatty acids, antioxidants and phytosterols which may contribute to prevention of heart disease and possible for cancer prevention (Giugliano et al., 2005).The importance of vegetable oils to the global economy becomes clear when considering the amount of vegetable oils produced and consumed worldwide. With exports and imports booming, strict control that aims to combat illegal attempts to boost profits is necessary.The increased attention has stimulated the interest in food authenticity. It covers many aspects including adulteration, mislabeling, characterization and misleading origin (Che Man et al., 2005). As in the global economy, traceability can be defined as the ability to track any food , feed, food-producing animal or substance that will be used for consumption, along all steps of production, processing and distribution (Costa et al., 2012).

Various vegetable oils have been reported to be adulterated, as adulteration of oil and fats falls into two main ways ; vegetable oils adulterated with cheaper vegetable oils or vegetable oils adulterated with animal fats. For example, olive oil and virgin olive oil which are the two most expensive oils compared to other edible oils (Al-Ismail, 2010) were commonly adulterated with hazelnut oil, sunflower oil and soybean oils (Christopoulou et al., 2002 ; Gamazo-Vazquez, 2003; Oussama, 2012). Although the adulteration of vegetable oils does not pose a threat to the consumer's healthy, in the specific cases of adulteration of olive oil with hazelnut may possess potential food allergenic which lead to life threating risk.

Whilst, lard contamination was detected in vegetable oil namely palm oil, palm kernel oil, canola oil as well in refined, bleached, deodorized palm olein (Marikkar et al., 2005 & Che Man et al., 2005). Pork and lard in food are serious matters from a religious point of view such as Islam as their religion prohibits their followers from consuming any food containing pork or lard (Che Man et al., 2005). The high content of saturated fatty acids and low proportion of polyunsaturated fatty acids in animal fats has major health risk factor, resulting in coronary heart disease (Marikkar et al., 2003).

There are several ways in which food can be mislabeled or characterized by including the substitution of one ingredient by a similar but cheaper one and over-declaring a quantitative ingredient (Gimenez et al., 2010). Also, the quality and uniqueness of edible vegetable oils namely virgin olive oil is a result of the different cultivar, environment and cultural practices. Thus, quality of the product is closely linked to its geographical origin and therefore the subject of adulteration is based on the mixing of oils from one region with those of lower quality grades for the case of virgin olive oil (Cosio et al., 2006). This practices is a commercial exploitation of olive oil and causes major loss in economic value, it has been estimated a loss of 4 million euros per year for countries in the Europen Union due to this activities(Zabaras et al., 2004).

Relatively, adulteration doesn't only focus on the highest commercial value product such as olive oil and virgin olive oil. Virgin coconut oil, a newcomer in oils and fats markets was also counterfeited with palm kernel oil (Manaf et al., 2006, Rohman et al., 2009 & 2010). Adulteration practices have also extended to other edible oils such as Cod Liver and Red Fruits (Pandanus conoideus) plant in the pandan family. Cod Liver oil has been a focus of a growing interest due to its nutritional advantages. It contains high levels of long chains n-3 fatty acids cis-5,8,11,14,17- eicosapentaenoic (EPA) and cis-4,7,10,13,16,19-docosahexaenoic (DHA) which are believed to play a preventive role in cardiovascular disease and in the alleviation of other health problems (Rohman and Che Man, 2009). Cod Liver oil was adulterated with canola, corn, soybean and walnut oil which have a similar color which therefore makes it difficult to detect using naked eyes (Rohman and Che Man, 2011). Similar to Cod Liver oil, Red fruits which provides several treatments for degenerative disease such as cancer was adulterated with common edible vegetable oils with similar fatty acid profiles such as canola and rice bran oil (Rohman et al.,2010)

As edible vegetable oils market expands, their authenticity has become an important subject from both commercial and health perspective. From the legislative point of view, quality standards have been established through the requirement of quality labels that specify the chemical composition of each product. In olive oil, the International Olive Oil Council (IOOC) has introduced methods and limits for the trade standard in olive oil. Among the established methods for determining the authenticity of olive oils were by determining fatty acids andtriacylglycerol composition (Christopoulou et al., 2004; Mariani et al.,2006; Monfreda et al., 2012; Ruiz-Samblas et al., 2012). From the economical point of view, product authentication is essential to avoid unfair competition that can create a destabilized market and disrupt the regional economy and even the national economy (Cordella, 2002). This is done by selling adulterated products at below the cost of the pure production which can drive producers and packers to be out of business. Without direct evidence of adulteration, these impacts result in bad economic growth as well as reduce consumer's confidence towards the product (Fairchild et al., 2003).

Similar to the common edible oils such as olive oil, virgin olive oil and virgin coconut, in recent years there has been rising production (supply) and consumption (demand) of crude palm oil. The demand for domestic consumption has gradually increased with a rather slow growth in production (Ramli , 2011). This widening gap between demand and production thus leads to frauds or adulteration. In Nigeria, the adulteration is believed to be practiced by producers in order to increase the quantity of crude palm oil (CPO), for the sole purpose of profit maximization (Okogeri, 2013). The adulteration practice is normally done without considering its possible effect on the quality of palm oil and the health of consumers. Red dye commonly used in the coloring of leather red, coloring of clothes, calabashes and as a body pigment is added into crude palm oil. This additive is to improve the colour and thus increase the market value without knowing the effect of the red dye on consumers health (Okonkwa et al., 2010; Okogeri,2013).

At present in Malaysia, there is no published investigation of adulteration of crude palm oil reported to our best knowledge. However, possible adulterants which could be used include sludge oil and used cooking oil (from deep frying) which were added into crude palm oil due to their abundance supply and similarity to the genuine oil (author, personal communication). The oil palm is a moocotyledon belonging to the genus Elaeis. It is a perennial tree crop and the highest oil producing plant which yield averagely at 3.7 tonnes of oil per hectare per year in Malaysia (Sundram,2001). The crop is unique in that it produces two types of oil. The fleshy mesocarp produces palm oil which is used mainly for its edible properties and the kernel produces palm kernel oil which has wide application in the oleochemical industry. Currently, Malaysia and Indonesia produce about 85% of world’s crude palm oil.

Extraction of crude palm oil (CPO) from fresh fruit bunches require steam for sterilization and water for dilution, which finally contribute to substantial amounts of water being discharged as sludge oil or palm oil mill effluent (POME) (Wafti et al., 2010). This generated about 60% of sludge oil for every tonne of fresh fruit bunches (FFB) processed in mills. As for recycled oil, countries in Asia such as China, Malaysia, Indonesia, Thailand, Hong Kong, and India produces about 40,000 tonnes per year of used cooking oil or waste cooking oil (Ismail, 2005). Improper waste management of used cooking oil leads to discharge to environment which leads to environment pollution or returned to human for consumption through food chains (Hanisah et al., 2013).

The increased demand for edible food oil caused entailed shortages of materials. Consequently, to fulfill the demand, common type of edible food oils were adulterated with cheaper materials as well as to improve the visual appearance of impure food oil. Despite the existence of food regulations which ensure that the standards are fulfilled, the constant evolution of adulteration practices have resulted in continuous amendment of the food regulation over time. As customers’ concern about the quality and safety of the edible oil increases, significant progress in natural science and analytical science were established to ensure food quality. Advances in analytical chemistry provided knowledge about the composition and properties of food which could be used to differentiate between genuine and adulterated products. In detecting adulteration in olive oil, analytical chemistry instrument such as chromatograph, thermal and spectral analysis were used to characterize the chemical compositions of olive oil and its adulterants, this analytical method is adapted in these studies.

Chromatographic techniques consist of gas and liquid chromatography which analyses are based on the separation of components. Advantages of chromatographic technology are its high separation efficiency and most accurate quantify results (Zhang et al.,2011). Whereby, thermal analysis by differential scanning calorimetry (DSC) allows the physical change that occur upon heating and cooling were able to be determined. These technique possesses the advantages of being relatively quick and simply to carry out with minimal sample preparation (Marikkar et al., 2003). Similarly by spectroscopy techniques, samples are analyses rapidly and non-destructively. Information about the molecular bonds present and therefore give details of the types of molecules present in the samples. These techniques provides a relatively low financial cost of obtaining and running the instrument (Reid et al., 2006).

Common edible oils such as olive oil and virgin coconut oil are the primary targets for adulteration in European countries. In the countries at tropical belt of Africa, Southeast Asia and parts of Brazil, crude palm oil is a main commodity to be adulterated. With an increase in demand for crude palm oil in these regions, it is not surprising that attempts by some devious suppliers to maximize revenues by counterfeiting and adulterating practices are conducted. Taken that, the main objectives of this project is to determine adulteration of crude palm oil by characterisation of the physiochemical properties of crude palm oil and its adulterant. To achieve the main objectives of the project, specific studies were designed as following :

i. To investigate current quality quantification by Free Fatty Acid, FFA and Deterioration of Bleachability Index, DOBI is able to detect adulteration.

ii. To characterize the crude palm oil, adulterant and admixtures of crude palm oil and adulterant via chromatographic techniques.

iii. To characterize the crude palm oil, adulterant and admixtures of crude palm oil and adulterant via thermal behaviour based on the crystallization and melting profiles.

iv. To characterize the molecular absorption and transmission of crude palm oil, adulterant and admixtures of crude palm oil and adulterant via fourier transform infrared (FTIR)

v. To process obtain results by multivariate analysis and regression modelling to quantify concentration of adulterant in crude palm oil blends.

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