OCCURRENCE AND EVALUATION OF HEAT GENERATED TOXICANTS IN REFINED PALM OILS AND FRYING OF SELECTED OILS

By

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June 2021

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The 2-monochloropropane-1,3-diol esters (2-MCPDE), 3-monochloropropane-1,2-diol esters (3-MCPDE) and glycidyl esters (GE) are contaminants formed during refining process, whilst acrylamide developed during heat treatment of carbohydrate-rich products. This study aimed to investigate the occurrence and development of these contaminants during refining of palm oil and to evaluate the effect of carbohydrate-based and protein-based foods during intermittent frying process. This study also aimed to optimize a Nuclear Magnetic Resonance (NMR) method for detection of these contaminants in vegetable oils and fried foods. Analyses on 68 samples of palm oil products showed that the 2-MCPDE, 3-MCPDE and GE concentrations were the lowest in crude oil (below LOD), and highest in RBD olein (> 6.0 mg kg^{-1} for 3-MCPDE and > 20 mg kg^{-1} for GE). Chloride content analysis on the CPO samples showed huge variations of the content due to environmental and processing factors, as well as quality of fresh fruit bunches (FFB).

Frying experiments using batch open fryers were carried out using palm olein, sunflower, canola and soybean oils. Frying oils were subjected to 2-MCPDE, 3-MCPDE and GE analyses, along with the fried foods which were subjected to acrylamide analysis. It was observed that there was a significant reduction in the 2-MCPDE, 3-MCPDE and GE content in all oils. Sodium chloride content in the fried food was also found to affect the formation of 3-MCPDE in the frying oils. As for the acrylamide, the content in both French fries and chicken nugget was observed to increase gradually as frying progressed with higher content in the French fries compared to chicken nugget.

The NMR method for the detection of 3-MCPDE and GE in frying oils was successfully optimized using deuterated chloroform. The chemical shifts of the

standards and samples for these two compounds were successfully identified at δ_H 0.86, 1.25, 1.60, 2.32, 3.64, 4.20, 4.33 and 5.21 for 3-MCPDE. For 2-MCPDE, the chemical shifts were detected at δ_H 0.86, 1.26, 1.61, 2.33, 4.22 and 4.29; whilst GE was detected at δ_H 0.86, 1.26, 2.33, 2.63, 2.83, 3.19, 3.90 and 4.40. The NMR method for acrylamide in fried foods has also been successfully optimized using deuterated water. The chemical shifts of the acrylamide standard detected were at δ_H 5.64, 5.66, 6.04, 6.07, 6.11 and 6.15.

The palm oil refiners are encouraged to take immediate mitigation actions in order to reduce the formation of 3-MCPDE and GE in the refined palm oil products. This is crucial to ensure Malaysian palm oil products contain 3-MCPDE and GE below the regulatory limits which are 2.5 mg kg^{-1} for 3-MCPDE and 1.0 mg kg^{-1} for GE. This can be achieved by optimizing the process conditions during the refining process, as well as washing of CPO prior to the refining to remove the precursors in the formation of these compounds. It is also necessary to maintain good quality of refined oils in order to control the formation of these compounds in the downstream products. In addition, NMR technique could be recommended as a quality control instrument for 3-MCPDE and GE content in the refined oil in comparison with GC-MS technique.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KEJADIAN DAN PENILAIAN BAHAN TOKSIK TERHASIL DARIPADA HABA DI DALAM MINYAK KELAPA SAWIT TERTAPIS DAN PENGGORENGAN MINYAK TERPILIH

Oleh

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Ester 2-monochloropropane-1,3-diol (2-MCPDE), ester 3-monochloropropane-1,2-diol (3-MCPDE) dan ester glisidil (GE) adalah bahan cemar yang terbentuk semasa proses penapisan minyak, manakala akrilamida terbentuk semasa proses haba bagi produk kaya dengan karbohidrat. Kajian ini dijalankan untuk mengesan pembentukan bahan cemar ini semasa proses penapisan minyak sawit dan untuk menilai kesan makanan berasaskan karbohidrat dan protein semasa proses penggorengan sekejap. Kajian ini juga bertujuan untuk mengoptimumkan kaedah Resonans Magnetik Nuklear (NMR) untuk pengesanan bahan cemar ini dalam minyak sayuran dan makanan bergoreng. Analisis ke atas 68 sampel produk minyak sawit menunjukkan bahawa kandungan 2-MCPDE, 3-MCPDE dan GE adalah yang paling rendah dalam minyak mentah (di bawah LOD), dan tertinggi dalam RBD olein (> 6.0 mg kg^{-1} untuk 3-MCPDE dan > 20 mg kg^{-1} untuk GE). Analisa kandungan klorida pada sampel minyak sawit mentah menunjukkan variasi kandungan yang besar disebabkan oleh faktor persekitaran dan pemprosesan, serta kualiti buah tandan segar (FFB).

Eksperimen penggorengan terbuka berkelompok telah dijalankan menggunakan minyak olein sawit, bunga matahari, kanola dan minyak kacang soya. Minyak penggorengan telah dianalisa kandungan 2-MCPDE, 3-MCPDE dan GE, manakala produk makanan yang digoreng telah dianalisa kandungan akrilamida. Didapati bahawa terdapat pengurangan ketara dalam kandungan 2-MCPDE, 3-MCPDE dan GE dalam kesemua minyak. Kandungan natrium klorida dalam makanan yang digoreng juga mempengaruhi pembentukan 3-MCPDE dalam minyak goreng. Bagi akrilamida pula, kandungan dalam kedua-dua kentang goreng dan nugget ayam diperhatikan meningkat secara beransur-ansur apabila proses penggorengan dengan kandungan yang lebih tinggi dalam kentang goreng berbanding nugget ayam.

Kajian terakhir adalah untuk mengesan kandungan 2-MCPDE, 3-MCPDE dan GE di dalam minyak sayuran serta kandungan akrilamida dalam produk makanan menggunakan peralatan NMR. Kaedah pengesanan telah berjaya dioptimumkan menggunakan *deuterated* klorofom sebagai pelarut, di mana peralihan kimia untuk standad dan sampel bagi kesemua bahan kontaminan telah berjaya dikesan pada δ_H 0.86, 1.25, 1.60, 2.32, 3.64, 4.20, 4.33 dan 5.21 untuk 3-MCPDE. Manakala bagi 2-MCPDE, peralihan kimia telah dikesan pada δ_H 0.86, 1.26, 1.61, 2.33, 4.22 dan 4.29; sementara GE telah dikesan pada δ_H 0.86, 1.26, 2.33, 2.63, 2.83, 3.19, 3.90 dan 4.40. Kaedah pengesanan untuk akrilamida juga telah berjaya dioptimumkan menggunakan pelarut *deuterated* air di dalam bahan larutan piawai dan produk makanan. Peralihan kimia bagi akrilamida telah berjaya dikesan pada δ_H 5.64, 5.66, 6.04, 6.07, 6.11 dan 6.15.

Kilang minyak sawit digalakkan untuk mengambil tindakan mitigasi segera untuk mengurangkan pembentukan ester 3-MCPD dan ester glisidol dalam produk minyak sawit tertapis. Ini bagi memastikan produk minyak sawit Malaysia mengandungi ester 3-MCPD dan ester glisidol di bawah had yang telah ditetapkan, iaitu 2.5 mg kg^{-1} bagi ester 3-MCPD dan 1.0 mg kg^{-1} bagi ester glisidol. Ini dapat dicapai dengan mengoptimumkan proses penapisan dan penulenan, serta mencuci minyak sawit mentah bersama air sebelum dihantar untuk proses penapisan bagi mengurangkan pembentukan sebatian ester 3-MCPD ini. Ia juga perlu untuk menjaga kualiti minyak tertapis ini bagi mengelakkan pembentukan sebatian ester ini semasa pengeluaran produk hiliran. Di samping itu, teknik NMR juga dapat digunakan sebagai teknik pengukuran bagi mengawal pembentukan sebatian ester 3-MCPD dan ester glisidol ini di dalam minyak masak tertapis sebagai perbandingan dengan teknik GC-MS.

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I certify that a Thesis Examination Committee has met on (date of viva voce) to conduct the final examination of Raznim Arni Abd. Razak on her thesis entitled "The Occurrence and Evaluations of Heat Generated Toxicants in Refined Palm Oils and Frying of Selected Oils" 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 (insert the name of relevant degree).

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LIST OF ABBREVIATIONS

3-MCPD	3-monochloropropane-1,2-diol
3-MCPDE	3-monochloropropane-1,2-diol esters
2-MCPDE	2-monochloropropane-1,3-diol esters
GE	Glycidyl esters
RBD	Refined, Bleached, Deodorized
CPO	Crude palm oil
GC-MS	Gas chromatography mass spectrometry
NMR	Nuclear magnetic resonance
FFA	Free fatty acids
AOCS	American Oil Chemists' Society
AOAC	Association of Official Agricultural Chemists
ASTM	American Society for Testing and Materials
IUPAC	International Union of Pure and Applied Chemistry
CODEX	Codex Alimentarius Commission
EFSA	European Food Safety Authority
IARC	International Agency for Research on Cancer
JECFA	Joint Expert Committee on Food Additives
COP	Code of Practice
DAG	Diacylglycerol
TAG	Triacylglycerol
MAG	Monoacylglycerol
HPLC	High performance liquid chromatography
ELSD	Electron light scattering detector
PV	Peroxide value

AnV	p-Anisidine value
FAC	Fatty acid composition
FAME	Fatty acid methyl ester
OSI	Oil stability index
TPC	Total polar compounds
POo	Palm olein
SFO	Sunflower oil
SBO	Soybean oil
CAN	Canola oil
SFA	Saturated fatty acids
MUFA	Monounsaturated fatty acids
PUFA	Polyunsaturated fatty acids
TFA	Trans fatty acids

CHAPTER 1

INTRODUCTION

1.1 Background of study

Processing of palm oil includes milling, refining and fractionation processes. A typical palm oil mill operation unit comprises sterilization, stripping, digestion and pressing, clarification, purification, drying and storage. Palm oil mill produces crude palm oil and kernels as primary products and biomass as secondary products. After the milling process, the crude oils are refined to remove undesirable substances. During refining, minor components including oxidation products, free fatty acids (FFAs), phospholipids, pigments, trace metals and other impurities are removed (Ramli et al., 2011). After the refining process, the refined oil undergoes fractionation stage whereby refined olein (liquid phase) and refined stearin (solid phase) are produced.

Food safety issues related to process-developed contaminants such as 3-monochloropropane-1,2-diol esters (3-MCPDE) and glycidyl esters (GE) in edible oils was first identified in 2004. The first publication on the detection of 3-MCPDE in various food products (e.g. crisp bread, salty crackers, potato crisps, French fries and pickled olives) was reported by a group of researchers from the Czech Republic (Divinová et al., 2004; Svejkovská et al., 2004). Since then, numerous publications reported on the occurrence of these compounds especially in refined vegetable oils and fats (Kuhlmann, 2011; Razak et al., 2012; Weiβhaar and Perz, 2010). Formation of 3-MCPDE has been linked to the reaction of precursor (chloride) at high temperature (above 260°C) during deodorization stage of oil refining process (Ramli et al, 2015). GE formation was mainly due to high temperature during deodorization stage too (Cheng et al., 2017). In May 2016, the European Food Safety Authority (EFSA) published a report on the levels of 3-MCPDE and GE in various foodstuffs as well as in refined oils and fats, which was based on their survey and previous data reported by other researchers. In the report, the levels of these compounds were significantly higher in refined palm olein compared to the other oils. This has triggered a concern among the palm oil producers especially in Malaysia wherein leading to various mitigation actions identified since then to tackle this issue.

The refined oils are used as a frying medium for cooking food products. Oils that are normally used as frying medium are palm olein, sunflower oil and soybean oil. During the frying process, the food was brought in direct contact with hot oil whereby the food surface becomes golden yellow to dark brown and developed a pleasant fried food flavor (Berger, 2005; Ismail, 2005; Tarmizi and Niranjan, 2013). Among the common fried foods are potato chip, nugget and egg. Depending on the food, frying oils or fat penetration varies in degrees contribute to richness, lubricity, flavor and calories. Frying techniques depend on the amount of oil or fat required, cooking time, type of cooking vessel and food

composition.

Besides 3-MCPDE and GE, acrylamide is also a heat-induced contaminant which was first reported in 2002 by Swedish National Food Administration in a wide range of fried and oven-baked products (Löfstedt, 2003). Acrylamide is formed during Maillard (browning) reaction (Stadler et al., 2002) involving asparagine and reducing sugars, which influences the quality of the food (i.e. flavor, color and aroma) (Vinci et al., 2012). In addition, acrylamide can also be formed via ammonia and acrolein in the absence of asparagine, through lipid degradation pathway (Yasuhara et al., 2003).

In relations to toxicological assessment, 3-MCPD is categorized as “possible human carcinogens” (Group 2B); whilst glycidol and acrylamide are categorized as “probably carcinogenic to humans” (Group 2A) by the International Agency for Research on Cancer (IARC) (IARC, 1994 and 2000). Based on this safety and health concerns, this study was carried out to identify the occurrence of 3-MCPDE and GE in palm oil during refining and fractionation process collected from selected refineries located throughout Malaysia. These data will be used as part of mitigation plans for the Malaysian palm oil industry for the production of palm oils with low 3-MCPDE and GE. In order to tackle the downstream industry, this study was also proposed to look into the formation of 3-MCPDE and GE in the frying oils as well as acrylamide formation in the fried foods due to safety and health concern. In parallel of the study on the frying oils, acrylamide formation was also monitored on two different food products (French fries and chicken nuggets) which were used in the frying step.

In this work, quantification of 3-MCPDE, GE and acrylamide were carried out using gas chromatography with mass spectrometry (GC-MS). In addition, an optimized method was developed using nuclear magnetic resonance (NMR) technique to qualify and quantify the presence of 3-MCPDE and GE in the oils before and after frying, as well as acrylamide content in the fried foods. The advantages of NMR method are that it is a non-destructive technique which is able to identify and elucidate the structure of chemical compounds, and it provides a unique approach for analysis of oils and fats (Sherazi and Mahesar, 2015).

Even though few factors affecting the formation of 3-MCPDE and GE have been identified and reported by other researchers, most of the studies were carried out on samples prepared in a specific and controlled conditions. There are less or little information on the occurrence data especially in Malaysian palm oil refined products. Various studies have been conducted on the occurrence of 3-MCPDE, GE and acrylamide during deep-fat frying on specific food products (i.e. potato chips and French fries), and mostly were carried out using palm oil, palm olein, sunflower and rapeseed oils. The use of NMR method was set to enhance on the information of 3-MCPDE, GE and acrylamide quantification.

1.2 Problem statement

The data on the occurrence of heat-induced toxicants like 3-MCPDE and GE is important for the palm oil producers as well as regulators to prepare for future standard operating procedures (SOPs) in setting up the maximum levels for these contaminants. Recently, Codex Alimentarius Commission has established a Code of Practice (COP) for the reduction and mitigation of 3-MCPDE and GE in refined oils and fats. It is expected that the COP implementation will be in 2022 wherein Codex will establish the maximum levels for these compounds. The European Commission (EC) will impose two maximum levels (1.25 mg kg^{-1} for oils and fats from maize, coconut, rapeseed, sunflower, soybean and palm kernel and mixtures of oils and fats from this category) and 2.50 mg kg^{-1} for other vegetable oils and fish oil and mixtures of oils and fats from this category, effective by January 2021 (European Commission, 2018). However, the EC has already set the maximum level for GE at 1 mg kg^{-1} in all vegetable oils. In the case of acrylamide, the Joint Expert Committee on Food Additives (JECFA) proposed that intake of 1 and 4 µg kg^{-1} body weight per day could represent an average and high intake of acrylamide including children. In view of this, it is important for vegetable oils producers and food manufacturers to regularly monitor the formation of these contaminants during the oil and food processing to ensure that the levels are or will not exceed the available or proposed regulatory limits. As such, in order to continuously monitor the presence of these contaminants in the oils and food products, there is a need to optimize a faster and non-destructive technique such as NMR spectroscopy besides the current technique using GC-MS, which takes a longer analysis time.

1.3 Significance of study

The findings from this research will be the source of information to strategize the mitigation steps for the prevention of the formation of these contaminants especially in the refined or processed palm oils produced at different locations in Malaysia. A verified method for the establishment of Malaysian National Code of Practice is crucial in order to mitigate the level of these contaminants in Malaysian palm oil products. Moreover, data generated from this work would also offer better understanding on the formation of these contaminants during frying, which could lead to possible mitigation steps.

1.4 Hypotheses of study

It was hypothesized that:

i. Varying practices adopted at the palm oil mills and refineries at different locations may contribute to producing FFB which may contain the precursors of 3-MCPDE and GE in palm oil products.
ii. Intermittent frying of different cooking oils and oil quality parameters

affect the formation of 3-MCPDE and GE in the frying oils and acrylamide in the fried food.

iii. NMR method to quantify the 2-MCPDE, 3-MCPDE and GE in the frying media and acrylamide in the fried foods to possible assist in the quality analysis of the frying oils and fried foods.

1.5 Objectives of study

Hence, three objectives were proposed for this study in view to overcome the stated problems:

i. To study the occurrence of 3-MCPDE and GE in CPO and refined palm oil products produced from different locations in Malaysia as well as commercial palm products from Asian markets.
ii. To study the intermittent frying effect on quality parameters, 3-MCPDE, GE in frying oils and acrylamide formation in French fries and chicken nuggets using various types of cooking oils; and
iii. To utilize an NMR method for the qualitative and relative quantification of 3-MCPDE, GE and acrylamide in the frying oils and fried foods.

The content of this thesis is as follows:

Chapter 2 is a literature review section on the occurrence and formation, toxicity issues, mitigation approaches and analytical techniques for the analysis of 3-MCPDE, GE and acrylamide.

Chapter 3 is a study on the occurrence of 3-MCPDE and GE in CPO and in refined palm oils collected from different locations in Malaysia. The method of analysis is also verified based on the AOCS Method Cd 29a-13, and a cross-check was conducted between MPOB and European laboratories to look into the comparison of the results. In addition, determination of 3-MCPDE and GE were also carried out on commercial cooking oils from Malaysian and Asian markets. Effect of chloride content in CPO with the 3-MCPDE and GE levels in the refined palm oils were also being studied.

Chapter 4 is the intermittent frying effect on oil quality indices (moisture and oil content, peroxide, *p*-anisidine value and TOTOX values, free fatty acids, fatty acid composition, color, oil stability index and polar compound fractions), 3-MCPDE and GE contents in frying oils (palm olein, sunflower, soybean and canola), and acrylamide contents in fried foods (French fries and tempura chicken nuggets). Significant correlation analysis was also conducted to study the relationships between oil quality parameters and the 3-MCPDE, GE and acrylamide contents during the frying process.

Chapter 5 is the utilization of NMR technique for the qualitative and relative quantification of 3-MCPDE and GE in the frying oils as well as acrylamide content in the fried foods.

Chapter 6 is the overall summary and conclusions of the thesis as well as recommendations for future works.

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