UNIVERSITI TEKNOLOGI MARA

A METHOD VALIDATION OF MODIFIED PRESSURIZED LIQUID EXTRACTION - GAS CHROMATOGRAPHY-MASS SPECTROMETRY FOR THE DETERMINATION OF 3- AND 2-MONOCHLOROPROPANEDIOLS FATTY ACID ESTERS AND GLYCIDOL ESTERS IN SELECTED FOOD PRODUCTS

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MSc

October 2021

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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Thesis Title	:	A Method Validation of Modified Pressurized Liquid Extraction - Gas Chromatography-Mass Spectrometry for The Determination of 3- and 2-Monochloropropanediols Fatty Acid Esters and Glycidol Esters in Selected Food Products
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ABSTRACT

The aim of the study is to establish the detection method for 3-, 2-monochloropropanediols Fatty Acid Esters (MCPDE) and glycidol Esters (GE) using modified pressurized liquid extraction (PLE) and gas chromatography mass-spectrometry (GC-MS). The PLE was adopted and modified (duration time of heating and number of static cycles) for reduced solvent volume and process time (to extract lipid from chocolate spread, infant formula, potato chips and sweetened creamer. The solvent selected for PLE was a mixture of iso-hexane and acetone at 100 °C with the lipid and analyte recovery ranging from 96.9% to 98.6% and 84.1% to 107.5%, respectively. The derivatization of analytes was adopted from American Oil Chemists' Society Official (AOCS) method Cd29a-13 for GC-MS analysis. The results showed that the coefficient of determination (R^2) of all analytes was > 0.99. The limit of detection (LOD) was 0.1 mg kg^{-1} expressed in lipid basis for both 3- and 2-MCPDE, and 0.2 mg kg^{-1} expressed in lipid basis for GE. The limit of quantitation (LOQ) was 0.3 mg kg^{-1} expressed in lipid basis for both 3- and 2-MCPDE, and 0.6 mg kg^{-1} expressed in lipid basis for GE. A blank spiked with 3- and 2-MCPDE (0.3, 2.1 and 7.2 mg kg^{-1}) and GE (0.6, 4.7 and 16.6 mg kg^{-1}) were chosen for accuracy and precision tests. The recoveries were between the range of 91.7% to 105.9%. Both repeatability and within-laboratory reproducibility of the analysis were within the acceptable level of precision ranging from 1.7% to 16%. This is the first time that full validation procedure extending to both accuracy and precision tests was carried out for sweetened creamer and chocolate spread. Overall, the combined protocol of modified PLE and AOCS Cd29a-13 was successfully validated for both solid and liquid food samples with lipid content from 10% to 30%. A validated method was applied to determine the concentration of 3-, 2- MCPDE and GE in chocolate spread, evaporated creamer, sweetened creamer, milk product and potato chips using modified Pressurised Liquid Extraction -Gas Chromatography-Mass Spectrometry. A total of 56 samples was selected from the retail market in Malaysian. The levels of 3-, 2- MCPDE and GE ranged from 0.1 mg kg^{-1} to 4.84 mg kg^{-1}. Chocolate spread was found to contain the highest concentration of 3- and 2- MCPDE at 4.84 mg kg^{-1} and 3.37 mg kg^{-1}, respectively. Meanwhile, potato chips contained the highest amount of GE at 4.07 mg kg^{-1}. The principal component analysis (PCA) showed that the 3-, 2- MCPD and glycidol contents was negatively correlated with fatty acid C16:0 (palmitic acid) and C18:0 (stearic acid).

ACKNOWLEDGEMENT

In the name of Allah, the Most Beneficent, the Most Merciful.

First of all, I would like to thank Allah for giving me the opportunity to embark on my Msc and for completing this long and challenging journey successfully.

I would like to express my deepest and sincerest appreciation to my supervisor, Dr. Adi Bin Md. Sikin for his valuable advises, patient and encouragement throughout this thesis study. His patience and passion in sharing his knowledge and experience by giving advices and good encouragement lead me to completion of this thesis. My gratitude and thanks also go to my co-supervisor, Dr. Azmil Haizam Bin Ahmad Tarmizi for his valuable advices and guidance. This work could not be complete without all of his kind assistance.

I am grateful to the Food Safety and Quality Division, Ministry of Health Malaysia for funding this research work. I would like to thanks to Pn. Zawiyah Binti Shariff and Pn. Nurhazwani Binti Abd Rahman for giving me helpful opinions and support during the entire study. My special gratitude is also dedicated to the Food Safety and Quality Laboratory State of Selangor and National Public Health Laboratory, Ministry of Health Malaysia for permission to use the facilities which made my research successful and meaningful. Appreciation was also extended to Faculty of Applied Sciences, Universiti Teknologi MARA for technical support.

Furthermore, special thanks to my colleagues and friends for their willingness to help and precious advices during my laboratory work and entire study. I would like to thanks Pn. Raznim Arni Binti Abdul Razak and Pn. Asimah from Innovative Products Laboratory, Malaysia Palm Oil Board for their help and guidance on fatty acid composition analysis. My sincere appreciation also goes to Miss May Yen and Mr. Tan from Shimadzu Malaysia for her guidance on GC-MS and Mr. Leong from Dionex Malaysia for the technical support on PLE.

Most of all, I dedicated my deepest love to the all my lovely family especially my husband, my mother, my father and my kids for their endless support, motivation and understanding throughout my study.

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