Ain Shams University

Faculty of Science

Determination of Polycyclic Aromatic Hydrocarbons in food using Gas Chromatography equipped with mass spectrometry

Presented by

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Thesis presented by

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ABSTRACT

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ABSTRACT

Name: Ahmed Hassan Ali Sayed

Title of the thesis: Determination of Polycyclic Aromatic Hydrocarbons in food using Gas Chromatography equipped with mass spectrometry

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Polycyclic aromatic hydrocarbons (PAHs) are a large group of organic compounds that are included in the European Union (EU) and US Environmental Protection Agency (US EPA) priority pollutant list due to their mutagenic and carcinogenic properties. Cooking and food processing at high temperatures have been shown to generate various kinds of genotoxic substances or cooking toxicants including PAHs.

A gas chromatography equipped with mass spectrometer (GCMS) method was developed and validated for determination of 16 polycyclic aromatic hydrocarbons (PAHs) in fish using modified quick, easy, cheap, effective, rugged, and safe (QuEChERS) method for extraction and solid phase extraction for sample cleanup.

PAHs were separated on a GCMS with HP-5msUltra Inert GC Column(30m, 0.25 mm, and 0.25 μm).Mean recovery ranged from56 to 115%.The extraction efficiency was consistent over the entire range where Indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene showed recovery (65, 69%), respectively, at 2 μg/kg. No significant dispersion of results was observed for the

other remaining PAHs and recovery did not differ substantially, and at the lowest and the highest concentrations mean recovery and RSD% showed that most of PAHs were between 70% and 120% with RSD less than 10%. The measurement uncertainty is expressed as expanded uncertainty and in terms of relative standard deviation (at 95% confidence level) is ±12%. This method is suitable for laboratories engaged daily in routine analysis of a large number of samples.

Survey on the presence of polycyclic aromatic hydrocarbons in grilled meat and chicken found that benzo(a)pyrene concentration in charcoal grilled chicken ranged from 0.49-7.20µg/kg and 2.01 µg/kg mean concentration, while for charcoal grilled meat ranged from 2.42-4.48 µg/kg and 0.36 µg/kg mean concentration.

The summation of 16PAHs concentrations in term of Benzo(a)Pyrene ranged from 0.03-10.65 µg/kg and mean concentration 3.03 µg/kg for charcoal grilled chicken, while for charcoal grilled meat from 0.10 to 6.49 µg/kg and mean concentration 1.13 µg/kg. The results suggested using of electric grill instead of traditional charcoal grill in order to reduce the contamination of meat and chicken with PAHs where the level of contamination reduced by 100%. The dietary intake for sum of seven carcinogenic PAHs in term of Benzo(a)Pyrene exceed than the average value by 1.6 and 2.3 times for meat and chicken respectively.

Keywords:

PAHs, QuEChERS, GCMS, Dietary intake, Benzo(a) Pyrene, Smoked Fish, Grilled meat, Grilled chicken, Egypt.

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AIM OF THE PRESENT WORK

The present work aim to develop and optimize an analytical method including separation and determination of Polycyclic Aromatic Hydrocarbons (PAH) in food using gas chromatography equipped with mass spectrometry (GC-MSD), followed by survey of PAHs contamination levels in grilled meat, chicken and smoked fish in Giza governorate. Finally study the effect of processing methods on PAHs levels in grilled meat and chicken, and then calculate the estimated acceptable dietary intake (ADI) of PAHs in food.

Method development and optimization

For this purpose, the work divided into the following parts:

1. Develop of suitable GC temperature programs.
2. Develop of suitable mass spectrometer detector (MSD) parameters.
3. Optimize the GC separation parameters (Oven temperature program, gas flow, injection mode).
4. Optimize the MSD detection parameters.
5. Development and optimization of the analytical method for extraction and clean up, then validation using the following criteria:-
    a) Recovery test
    b) Limit of detection (LOD)
    c) Limit of quantitation (LOQ)
    d) Linearity (including standard and method linearity)
    e) Accuracy (expressed as trueness)
    f) Precision (including repeatability and within-laboratory reproducibility)
    g) Measurement Uncertainty

Survey of PAHs contamination levels

6. Investigation of accumulation of PAHs in grilled meat and chicken and Smokes fish in Giza governorate (Egypt)
7. Study the effect of processing methods on PAHs Levels in grilled meat and chicken.

Estimation of acceptable dietary intake (ADI)

8. PAHs fish, meat and chicken evaluation of the acceptable dietary intake (ADI) for using data resulting from monitoring.

ACN Acetonitrile

ADI Acceptable Dietary Intake

AOAC Association of Official Analytical Chemists

BaP Benzo(a)pyrene

C18 Octadecyl carbon chain bonded silica

CAC Codex Alimentarius Committee

CAS Chemical Abstracts Service

DLLME Dispersive Liquid-Liquid Micro Extraction

EDI Estimated Daily Intake

EC European Communities

EN European Union

EPA Environmental Protection Agency

EU European Union

FAO Food and Agricultural Organization

FDA Food and drug administration

GC Gas Chromatography

GEMS Global Environment Monitoring System

GPC Gel permeation chromatography

HF-LPME Hollow Fiber Liquid-Phase Micro Extraction

HPLC High Performance Liquid Chromatography

HSSE Headspace Sorptive Extraction

IARC International Agency for Research on Cancer

ISO International Organization for Standardization

IUPAC International Union of Pure and Applied Chemistry nomenclature

JECFA Joint FAO/WHO Expert Committee on Food Additives

LOD Limit of Detection

LOQ Limit of Quantification

MAE Microwave-Assisted Extraction