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**CHROMATOGRAPHIC ANALYSIS OF
PETROLEUM-BASED CONTAMINANTS IN
EDIBLE OILS AND OLEOCHEMICALS**

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ABSTRACT

The main objective of this work is to explore the potential of high-performance liquid chromatographic (HPLC) and gas chromatographic (GC) techniques for the determination of petroleum-based organic contaminants in vegetable oils and related products. In this research, 2 types of contaminants namely thermal heating fluids and diesel fuel, which are still the issues of interest to the palm oil trade, have been addressed.

The 3 types of thermal heating fluids selected in this study were (1) the eutectic mixtures of diphenyl oxide and biphenyl, (2) partially hydrogenated terphenyls, and (3) synthetic hydrocarbons. It was found that by coupling a fluorescence detector to a HPLC system, the determination of the eutectic mixtures in vegetable oil and basic oleochemical samples could be carried out directly without any need of a pre-treatment step before the sample solutions were injected onto a reversed-phase HPLC column. This method has also been validated through an interlaboratory study, and accepted by the American Oil Chemists' Society (AOCS) as Recommended Practice Cd 25a-00.

Even though the direct HPLC method was good for the analysis of the partially hydrogenated terphenyls in oleochemical samples, saponification of vegetable oils was required for removing the interfering fluorescent components prior to analysis.

In order to determine the synthetic hydrocarbons in vegetable oil and basic oleochemical samples, a preparative column chromatography was incorporated to concentrate the fluid before the extract was subjected to further analysis. The study

showed that while reversed-phase HPLC with fluorescence detection is suitable for oleochemical samples, the quantitative analysis of the synthetic hydrocarbons in vegetable oils could only be carried out by GC due to the presence of interfering fluorescent components that co-eluted with the vegetable oil extracts. Finally, the determination of diesel fuel in vegetable oil samples was achieved by using a normal-phase HPLC with fluorescence detection.

All the methods developed are relatively simple, which are ideal for quality assurance purposes in the palm oil industry.

ABSTRAK

Tujuan utama kajian ini adalah untuk menguji potensi teknik HPLC dan GC dalam menentukan kehadiran bahan pencemar organik berasaskan petroleum di dalam minyak sayuran dan hasil berkaitan. Dua jenis bahan pencemar, iaitu bendalir pemanas terma dan bahan bakar disel yang diberi perhatian buat masa ini, telah dijadikan bahan penyelidikan.

3 jenis bendalir pemanas terma telah dipilih untuk kajian ini (1) campuran eutektik difenil oksida dan bifenil, (2) terfenil dihidrogenat secara separa, dan (3) hidrokarbon sintetik. Garbungan pengesan pendarfluor dengan sistem HPLC boleh menentukan campuran eutektik dalam minyak sayuran dan oleokimia asas secara terus tanpa melakukan sebarang langkah pra-rawatan sebelum sampel dimasukkan ke sistem HPLC fasa-terbalik. Di samping itu, kaedah HPLC ini telah pun diterima oleh AOCS sebagai *AOCS Recommended Practice Cd 25a-00*.

Walaupun kaedah HPLC juga sesuai untuk mengkaji terfenil separa dihidrogenat yang hadir didalam sebatian oleokimia, sampel minyak sayuran perlu disaponifikasikan untuk membuang komponen-komponen yang berpendarfluor yang boleh mengganggu analisis.

Bagi penentuan hidrokarbon sintetik dalam minyak sayuran dan oleokimia asas, kromatografi turus secara preparatif telah digunakan untuk memekatkan bendalir tersebut sebelum dibuat kajian lanjutan. Penemuan kajian menunjukkan HPLC fasa-terbalik dengan pengesan pendarfluor sesuai untuk sampel oleokimia. Sementara analisis kuantitatif bagi hidrokarbon sintetik dalam minyak sayuran boleh dilakukan dengan kaedah GC. Kaedah HPLC kurang sesuai bagi hidrokarbon

sintetik kerana kehadiran komponen berpendarfluor yang turut mengelusi bersama ekstrak minyak campuran.

Penentuan bahan bakar disel dalam minyak sayuran juga berjaya dilakukan melalui kaedah HPLC fasa-normal dengan pengesan pendarfluor.

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ABBREVIATIONS

AOAC	Association of Official Analytical Chemists' International
AOCS	American Oil Chemists' Society
CV	Coefficient of variations
ECL	Equivalent chain length
ELSD	Evaporative light scattering detector
FAME	Fatty acid methyl ester
FID	Flame ionization detector
GC	Gas chromatography
HPLC	High-performance liquid chromatography
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
LOD	Limit of detection
LOQ	Limit of quantitation
MIBK	Methyl isobutyl ketone
MPOB	Malaysian Palm Oil Board
MS	Mass spectrometry
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PORLA	Palm Oil Registration and Licensing Association
PORIM	Palm Oil Research Institute of Malaysia
r^2	Correlation coefficient
RI	Refractive index

SD	Standard deviation
TLC	Thin-layer chromatography
UV	Ultra-violet
VERNOF	Dutch Seed Crushers and Oil Processing Association