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Application News

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GCMS-QP2010 Ultra

Fingerprinting of Oils and Fats Using GC-MS in Combination with Chemometrics

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❑ Introduction

Oils and fats are widely used in the commercial manufacturing of food, preparation of fast food and domestic cooking as they enhance flavor and aroma of food. Thus the authentication of oils and fats is of paramount importance not only to consumers but also to manufacturers and local regulatory authorities. The consumers, on the other hand, are concerned with the type of the oils and fats that they consume as these oils and fats can affect their well-being. For example, the consumption of soya bean or peanut oil might trigger allergies like asthma, hives and atopic dermatitis in some consumers. In extreme situation, life-threatening and lethal anaphylaxis might result. Besides, research studies have reported that the prolonged consumption of saturated fats, such as lauric acid (C12:0), which exist mainly in animal fats will lead to an increase in the low-density lipoprotein (LDL) cholesterol in plasma. Besides nutritional and health benefits, religious beliefs also affect the choice of diet. For instance, vegetarians and Hindus are forbidden to eat food products which consist of animal fats and beef respectively. Likewise, Muslims and Jews have religious restriction on the consumption of porcine-originated food products. In view of the great demand for reliable techniques for the identification of various types of oils and fats, the feasibility of GC/MS with the combination of chemometrics in the differentiation of oils and fats was evaluated.

❑ Experimental

Five types of commercially available oils and fats of three different brands (Brand A, B and C) were purchased from local supermarkets. The vegetable oils included olive oil, canola oil, palm oil and soybean oil. The lard were obtained by heating the adipose tissues of pig purchased from three different local markets, at 90°C for 15 min. The fatty acids present in oils and fats were derivizatied to fatty acid methyl esters (FAMEs) using methanolic sodium hydroxide and boron fluoride in methanol (10% w/w).

Analysis was performed on using single quadrupole GCMS-QP2010 Ultra (Shimadzu Corporation) was employed in this work. The GC-MS conditions are shown in Table 1. Custom retention index mixture, which consisted of C7 to C32 hydrocarbon chains, were acquired from Restek (Bellefonte, USA) to generate the retention index for the GC/MS analysis of oils and fats.

The identification of the FAMEs was performed by matching both the peak's mass spectrum and retention index of the FAMEs to the GC/MS Metabolite Mass Spectral Database. All data were converted to the network Common Data Form (netCDF) files. The open source software, MZmine 2 (http://mzmine.sourceforge.net/) was employed for pre-processing of the raw GC/MS data.

Data analysis was performed using multivariate statistical analyses using SIMCA P+ version 12 software. (Umetrics, Sweden). The principal component analysis (PCA) was utilized for the discrimination of oils and fats.

Table 1: GC/MS Conditions for FAME Analysis

GC/MS	Shimadzu GC/MS-QP2010 Ultra system with AOC20i+s auto sampler
Injection mode	Split at Split ratio100:1
Injector volume	1.0 µL
Injector temperature	280 °C
Column	DB5-MS (30 m x 0.25 mmx 0.25 µm) (J&W Scientific)
Column oven program	40°C (2 min) – 6°C/min – 320°C (1 min)
Interface temp	250°C
Mode of ionization	Electron impact
Mass range	50-500 a.m.u.

Method Development

With the usage of a relatively non-polar capillary column, the FAMEs were chromatographically separated based on boiling points and polarities during GC/MS separation (**Figure 1**). The FAMEs was identified based on the mass spectra and the retention index generated with the hydrocarbon standard.

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FAMEs with longer alkyl chain and the least number of double bonds were eluted later.

For example, methyl palmitate (C16:0) eluted earlier at approximately 29.5 min as compared to the methyl stearate (C18:0) which eluted at 32.4 min from the GC column as observed in the chromatogram of peanut oil.

In addition, the elution order from the GC column was also dependent on the number of double bonds present as such the C18 FAMEs were found to be eluted in the order of C18:3, C18:2, C18:1and C18.

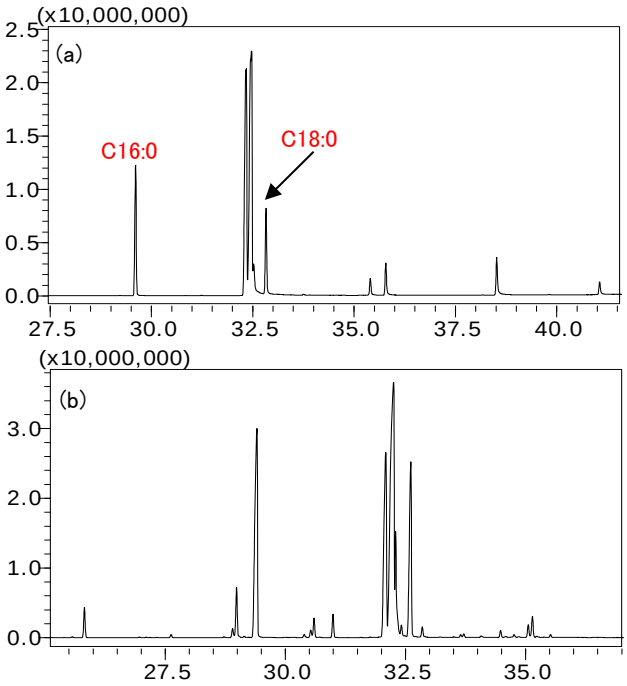

Figure 1: Examples of the GC/MS chromatograms of (a) peanut oil and (b) lard.

Discrimination between different types of oils and fats with exploratory PCA

With the vast amount of dataset obtained based on GC/MS analyses and their intrinsic similarities in the fingerprints, it was difficult to identify individual oil sample based on visual comparisons of the GC/MS fingerprints. Hence, principal component analysis(PCA) was employed.

The PCA score plot of GC/MS data in Figure 2 achieved excellent goodness of fit and validity (R2Xcumulative =0.943 and Q2Xcumulative= 0.907 which were close to 1. Examination of the scores plot indicated good experimental reproducibility since tight clustering of replicates for each type of oils and fats was observed.

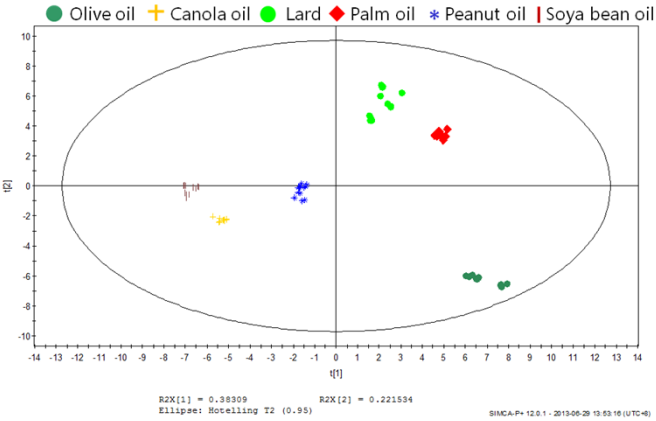

Figure 2: PCA score plot of the oils and fat based on GC/MS data.

It was also evident that grouping patterns according to the type of oils and fats can be observed demonstrating the feasibility of PCA model to distinguish their differences.

Lard and palm oil were located in the upper right quadrant of the score plots were differentiated from the rest of the oils and fats by methyl palmitate (mass fragments 74 and 87 at 29.5 min). On the other hand, vegetable oils such as soya bean oil and canola oil, which clustered in the negative quadrant of the score plot, showed higher percentage composition of methyl linoleate. (m/z 81 and 79 at 32.3 min)

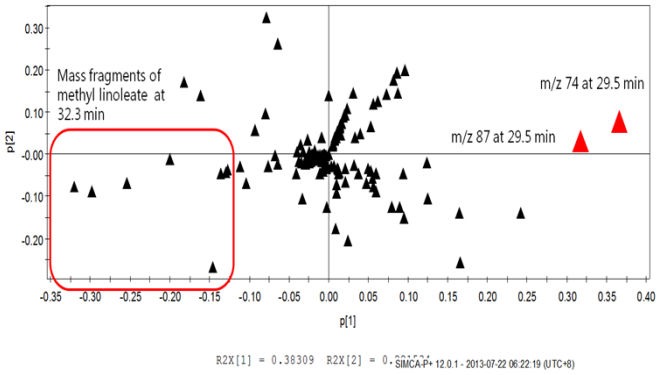

Figure 3: Loading plot of the oils and fat based on GC/MS data.

❑ Conclusions

Fingerprinting technique together with GC/MS and chemometrics were successfully developed for differentiation of different oils and fats, illustrating the possibility of using the methodology for the creation of a database for identification of oils and fats.

❑ References

1. Fang, G., Goh, J.Y., Tay, M., Lau, HF, Li , F.Y.S., Food Chemistry 138 146–1460 (2013).

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