SHORT COMMUNICATION

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CHEMICAL PROFILING OF PALM OIL AND OTHER SELECTED PLANT OILS BY PROTON NUCLEAR MAGNETIC RESONANCE COMBINED WITH CHEMOMETRICS

NOOR IDAYU TAHIR[1]*; ASIAH NABIHAN ISHAK[2]; TEH SOEK SIN[1]; ABRIZAH OTHMAN[1] and UMI SALAMAH RAMLI[1]

ABSTRACT

Nuclear magnetic resonance (NMR) spectroscopy is an important and universal tool for chemical profiling. Combined with chemometrics for statistical interpretation and visualisation, the technique has gained recognition as an efficient method for assessing the quality and safety of vegetable oils. Nevertheless, its application in palm oil traceability is scarce. In this investigation, different origins, types and preparations of palm oils, olive oils, virgin coconut oils (VCO) and seed oils from sesame, black caraway, argan, corn, peanut and sunflower were examined for their chemical attributes using proton (^1H) NMR. The analysis used milligrams quantity of samples and was proven to be rapid with minimal sample preparation and analysis time. The statistical components of the multivariate model generated from the dataset were significant and highlighted the characterisation of the specimens into groups of palm, olive, virgin coconut and seed oils based on the presence and abundance of various types of proton signals. Different packaging and blends of commercial palm cooking oils exhibited similar proton profiles while the lab-prepared palm oil is closely linked to VCO. The work contributes to the development of authentication and traceability analytical methods for palm oil and will be expanded into a predictive platform with a larger dataset.

Keywords: chemical signatures, NMR spectroscopy, oil purity and stability, proton signals, vegetable oil.

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INTRODUCTION

The oils of plant origin especially palm oil are largely used in the food-based application and oleochemicals (Parveez *et al*., 2021). In addition to being a renewable resource, each plant oil has its unique chemical, physical and nutritional properties, *i.e*., oils with high amounts of saturated fatty acids such as palm and coconut oils are well suited for frying due to their thermal stability (Mitrea *et al*., 2022), extra virgin olive oil (EVOO) and sesame oils show better oxidative stability due to their natural antioxidants (Hashempour-Baltork *et al*., 2016; Kittipongpittaya *et al*., 2020), while black caraway seed (*Nigella sativa*) oil contains fat-soluble bioactives which enriched and improved the stability of sunflower oil as a blend (Kiralan *et al*., 2017). Several plant oils of high value, *e.g*., EVOO are targets for adulteration with less expensive sunflower and corn oils, while some oils are purposely blended to achieve desirable criteria such as canola with palm oil to enhance ω-3 (omega) fatty acid (Hashempour-Baltork *et al*., 2016; Hong *et al*., 2017).

NMR spectroscopy is one of the reliable and widely used analytical tools to evaluate the purity and stability of edible oils (Almoselhy *et al*., 2014; Ravaglia *et al*., 2019) and to monitor oil compositional and oxidative changes (Alonso-Salces *et al*., 2021). Although it offers a high potential in detecting the chemical variability within food systems with

[1] Malaysian Palm Oil Board,
6 Persiaran Institusi, Bandar Baru Bangi,
43000 Kajang, Selangor, Malaysia.

[2] Department of Plant Science, Kulliyyah of Science,
International Islamic University Malaysia (IIUM),
Bandar Indera Mahkota, 25200 Kuantan, Pahang, Malaysia.

* *Corresponding author e-mail: idayu@mpob.gov.my*

minimal analysis time and sample preparation compared to gas chromatography that requires derivatisation, the technique is still underutilised mainly due to its high acquisition cost and the lack of instrument and data handling expertise by food scientists (Hatzakis, 2019). To facilitate the discovery of distinguishable chemical attributes from spectral patterns obtained from analytical instruments such as the NMR, a data analysis technique termed 'chemometrics' that links chemical information to interpretive models is required (Bressanello *et al*., 2018). Chemometrics determines the features that are statistically different between sample classes (Rankin *et al*., 2014) and enables characterisation and interpretation of the data output (Ishak *et al*., 2021). The future of edible oil traceability and geographical authentication relies on the evolving chemometrics tool that can harmonise the output from diverse analytical platforms into a fingerprint database (Tahir *et al*., 2022).

Although widely used in the assessment of botanical sources for other vegetable oils especially olive (Ingallina *et al*., 2019; Ravaglia *et al*., 2019), the utilisation of combined NMR spectroscopy and chemometrics approach in determining the origin and traceability of palm oil is still scarce as reviewed by Ramli *et al.* (2020). Only recently, the method of NMR and chemometrics was applied to monitor the thermostability of palm oil as reported by Pinto *et al.* (2021). In view of this, palm oils of different processing, packaging and blend were subjected to NMR spectroscopy and chemometrics along with selected commercial vegetable oils to gain insights into their chemical signatures and discover their distinct characteristics. This work demonstrates the capacity of the employed method for palm oil traceability and authentication efforts, and the data serves as a groundwork for obtaining baseline information to detect the genuineness of edible plant oils, especially palm oil.

MATERIALS AND METHODS

Oil Samples

Palm oil samples from three ripe (20 to 22 weeks after anthesis) *tenera* oil palm fresh fruit bunches (FFB) sourced from MPOB planting trials in Johor, Terengganu and Teluk Intan were manually prepared in the lab. The FFBs were chopped into spikelets and placed into stainless steel round wire baskets of a Hirayama HICLAVE HVE-50 autoclave (Hirayama Manufacturing Corp., Japan) and sterilised at 100°C for 60 min. The sterilised fruitlets were detached and fitted into a container for a mechanical screw-pressing machine. The resulting liquid mixture was allowed to stand in an oven at 50°C to separate the oil from water, sludge and debris. The oil was collected and filtered through anhydrous sodium sulphate (ChemAR System, Classic Chemicals Sdn. Bhd., Malaysia) until the crystals were not clumped together indicating moisture traces have been removed. The oil was labelled as lab-prepared crude palm oil (LCPO1, 2 and 3).

A total of 35 commercial plant oil samples from bottled and pouch-packed palm cooking oils, red palm oils, olive and extra virgin olive, virgin coconut, corn, sunflower, peanut, sesame, argan and black caraway seed oils were obtained from various commercial sources. All the commercial samples had their country of origin or manufacturing listed in accordance with the information stated on their packaging and manufacturer's website. The argan tree is naturally endemic to Morocco and a few other countries with dryland such as Algeria and Tunisia (Gharby and Charrouf, 2022), therefore it is suggested that Brand 1 argan oil (sample Argan1) was processed in the manufacturing country but originated from a different location such as Morocco. More than three replicates were analysed for the palm oil commercial samples due to their availability in the local grocery stores. The countries of origin or production of the samples are indicated in *Table 1*.

Proton (^{1}H) NMR Spectroscopy Analysis

The oil sample chemical profiles were determined using a JNM-ECZ600R/S1 600 MHz NMR spectrometer (JEOL, Japan). 50 mg of oil was dissolved in 1.0 mL of deuterated chloroform ($CDCl_3$) (Merck KGaA, Germany) and transferred into standard 5 mm NMR tubes (Norell Inc., USA) for direct measurement. The one-dimensional proton spectra were acquired at a frequency of 600.1723 MHz at 14.09637 Tesla (T) magnetic field strength, with the observation nucleus (x_domain) set as 'proton', 5 ppm observation centre frequency (x_offset) and 15 ppm observation range (x_sweep). The pulse width used for the measurement (x_pulse) was 3.15 μs at a pulse angle of 45°, while the pulse delay time (relaxation_delay) was 5 s for rapid and routine 1D ^{1}H experiment.

Data Analysis

The Delta NMR Processing and Control Software v6.0 (JEOL Resonance Inc., Japan) was used to view, process and annotate the raw spectral data in terms of peak intensity and chemical shifts (δ), expressed in parts per million (ppm) relative to tetramethylsilane (TMS, $\delta = 0.00$). The intensity and shift information were tabulated into a comma separated values (.csv) format file using the Delta v6.0 'Chemometrics' function with baseline correction and tolerance referencing at 0.2 ppm and bucket integration width of 0.02 ppm. Chemometric analysis was performed

TABLE 1. PLANT OIL SAMPLES AND THEIR SOURCES

	Oil specimen	Analysis label	Origin/production
1	Crude palm oil from fresh fruit bunch plantation 1	LCPO1	MPOB laboratory preparation
2	Crude palm oil from fresh fruit bunch plantation 2	LCPO2	MPOB laboratory preparation
3	Crude palm oil from fresh fruit bunch plantation 3	LCPO3	MPOB laboratory preparation
4	Extra virgin olive oil Brand 1	EVOO1	Commercial, Tunisia
5	Extra virgin olive oil Brand 2	EVOO2	Commercial, Spain
6	Extra virgin olive oil Brand 3	EVOO3	Commercial, Italy
7	Olive oil Brand 1	OO1	Commercial, Spain
8	Olive oil Brand 2	OO2	Commercial, Spain
9	Olive oil Brand 3	OO3	Commercial, Italy
10	Virgin coconut oil Brand 1	VCO1	Commercial, Malaysia
11	Virgin coconut oil Brand 2	VCO2	Commercial, Turkey
12	Virgin coconut oil Brand 3	VCO3	Commercial, Malaysia
13	Pouch packed palm cooking oil Brand 1	Pouch1	Commercial, Malaysia
14	Pouch Brand 2	Pouch2	Commercial, Malaysia
15	Pouch Brand 3	Pouch3	Commercial, Malaysia
16	Bottled palm cooking oil Brand 1	BPO1	Commercial, Malaysia
17	Bottled palm cooking oil Brand 2	BPO2	Commercial, Malaysia
18	Bottled palm cooking oil Brand 3	BPO3	Commercial, Malaysia
19	Bottled palm cooking oil Brand 4	BPO4	Commercial, Malaysia
20	Bottled palm cooking oil Brand 5	BPO5	Commercial, Malaysia
21	Bottled palm cooking oil with unknown blend % of peanut and sesame oil Brand 1	BPOM1	Commercial, Malaysia
22	Bottled palm cooking oil with unknown blend % of peanut and sesame oil Brand 2	BPOM2	Commercial, Malaysia
23	Bottled palm cooking oil with unknown blend % of peanut and sesame oil Brand 3	BPOM3	Commercial, Malaysia
24	Bottled palm cooking oil with unknown blend % of peanut and sesame oil Brand 4	BPOM4	Commercial, Malaysia
25	Red palm oil Brand 1	RPO1	Commercial, Malaysia
26	Red palm oil Brand 2	RPO2	Commercial, Malaysia
27	Corn cooking oil Brand 1	Corn1	Commercial, Malaysia
28	Corn cooking oil Brand 2	Corn2	Commercial, Malaysia
29	Sunflower cooking oil	Sunflower	Commercial, Malaysia
30	Sesame oil Brand 1	Sesame1	Commercial, Malaysia
31	Sesame oil Brand 2	Sesame2	Commercial, Malaysia
32	Sesame oil Brand 3	Sesame3	Commercial, Malaysia
33	Argan seed oil Brand 1	Argan1	Commercial, Turkey
34	Argan seed oil Brand 2	Argan2	Commercial, Morocco
35	Peanut oil	Peanut	Commercial, Italy
36	Black caraway seed oil Brand 1	BC1	Commercial, Egypt
37	Black caraway seed oil Brand 2	BC2	Commercial, Malaysia
38	Black caraway seed oil Brand 3	BC3	Commercial, Turkey

using the online public MetaboAnalyst Version 5.0 (https://www.metaboanalyst.ca) with a coefficient of determination calculated via leave-one-out cross-validation (LOOCV) (Lee *et al*., 2018). Preprocessing of the data includes normalisation by sum and Pareto scaling (mean-centered and divided by the square root of the standard deviation of each variable) and permutation tests by 'between' and 'within' classes separation distance (B/W). The hierarchical clustering heatmap was constructed using analysis of variance (ANOVA), Minkowski distance measure and average clustering. The diagnostic statistics to

evaluate the performance of the machine learning algorithms for the multivariate analysis is based on Q2 and R2 values (R2 > Q2, and, the difference between R2 and Q2 < 0.2) as outlined by Kiralj and Ferreira (2009).

RESULTS AND DISCUSSION

The resulting multivariate model constructed from the ^{1}H NMR profiles showed excellent statistical components with an accuracy of 0.81579, R2 (goodness of fit) value of 0.90322 and Q2 (the predictive ability of the model) of 0.83894. The permutation test revealed its significance under possible rearrangements of the observed data points (*Table* 2). The probability of attaining a result at least as extreme as the test statistic with a true null hypothesis (exchangeable sample classes) is expressed in *p*-value. Lower *p*-values indicate that the features are not interchangeable and that the original classification is relevant with respect to the data (Knijnenburg *et al*., 2009).

The principal component analysis (PCA) scores plot (*Figure 1*) of the ^{1}H NMR data showed clear groupings of the oil samples respective to their classes. The accompanying biplot displayed the chemical shifts that contributed to the greatest distribution of the samples shown in the scores plot. VCO samples were set apart from the other oils on the upper segment of the plots followed by the LCPO proton profiles which were scored in between the VCO and the commercial palm oil specimens based on their higher abundance of 1.26 and 1.27 ppm proton signals corresponding to the methylene protons of fatty acid chains (-$(CH_2)_n$-) (Di Pietro *et al*., 2020). The scores of RPO and refined palm oils (cooking palm oils either sold in bottles or pouches, labelled as BPOs, BPOMs and Pouches) were plotted on a lower segment next to the olive oils with higher abundance of 1.24 and 1.27 ppm signals also for fatty acid chain methylene protons [-$(CH_2)_n$-], and 0.87 ppm signal for acyl chain methyl protons (-CH_3).

RPO1 score was observed separately from another red palm oil specimen RPO2 towards the seed oil group. Higher proton peak abundances were found at shift 0.93-0.96 ppm in RPO1 proton spectra compared to the other palm oil samples (*Figure* 2) indicating the notable presence of linolenic acid (Alexandri *et al*., 2017), possibly due to its blend with another type of plant oil. RPO1 is a commercial blend of red palm and canola (rapeseed) oils, which supports this claim.

Linolenic acid is an octadecatrienoic, polyunsaturated fatty acid occurring in plant oils as either or both the α-linolenic (ALA), an omega-3 (ω-3/n-3) and the γ-linolenic (GLA), an ω-6 (n-6). It is found in a significant amount of at least 7.8%-9.9% in soybean and rapeseed oils (Dubois *et al*., 2007) (*Table 3*).

TABLE 2. PERMUTATION TEST STATISTICS

Permutation numbers	Empirical *p*-value
20	$p < 0.05$
100	$p < 0.01$
1 000	$p < 0.001$
2 000	$p < 0.0005$

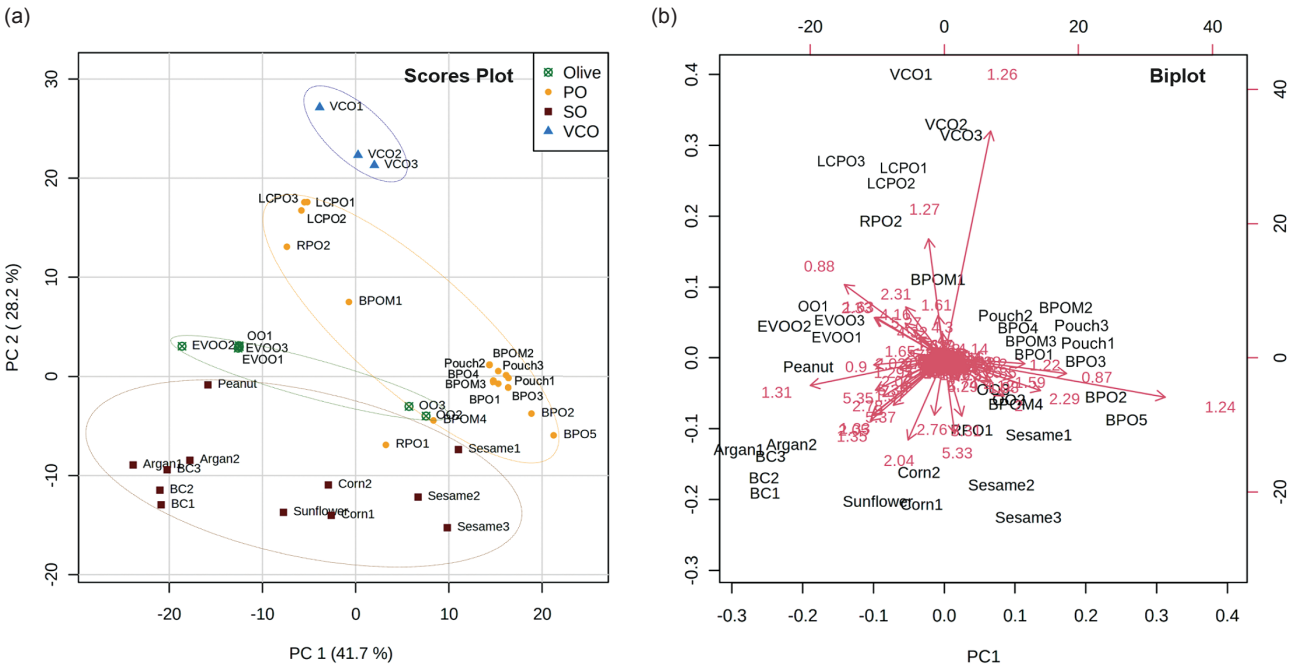

Figure 1. PCA scores plot (a) and biplot (b) of plant oils ^{1}H NMR data. Classes of oils: Olive and extra virgin olive oils (Olive); palm oil (PO); seed oils (SO) and virgin coconut oil (VCO).

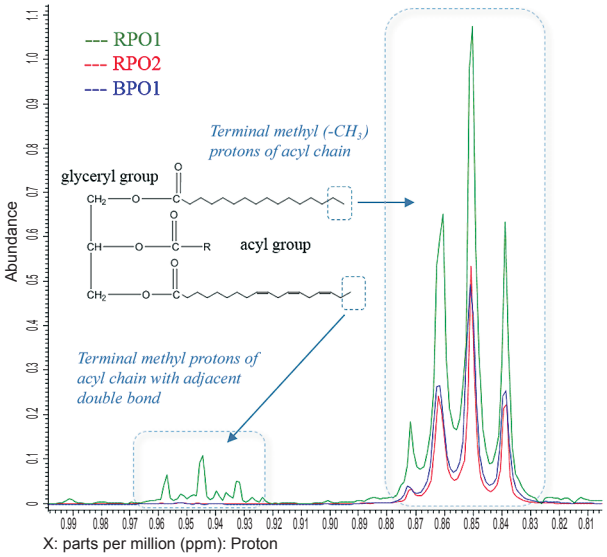

Figure 2. Traces of proton signals at shift 0.93-0.96 ppm in RPO1 proton spectra compared to RPO2 and BPO1 denoting the terminal methyl (-CH_3) group of linolenic acid.

TABLE 3. PERCENTAGE OF LINOLENIC ACID (18:3) CONTENT IN COMMERCIAL PLANT OILS

Oils	%	
	α (18:3 n-3)	γ (18:3 n-6)
Hemp seed oil	19.7	2.8
Rapeseed	9.9	0.0
Soybean	7.8	0.0
Black caraway	2.4	0.0
Argan	1.4	0.0
Corn	1.0	0.0
Olive	0.6	0.0
Sunflower	0.5	0.0
Sesame	0.4	0.0
Peanut	0.4	0.0
Palm	0.3	0.0
Coconut	0.1	0.0

Source: Dubois *et al.* (2007).

The abundance of the proton signal for the terminal methyl (-CH_3) group of linolenic acid which appears as a triplet after 0.9 ppm as shown in *Figure 2* was averaged and plotted into a column chart according to their respective species of black caraway, argan, corn, sunflower, sesame, peanut and coconut; the processing of EVOO and OO, and packaging of pouch-packed, bottled and lab-prepared crude palm oil (*Figure 3*). RPO1 showed the highest abundance of the triplet signal compared to the other oil specimens most likely attributed to its canola (rapeseed) oil blend.

The scores for EVOO and OO were observed in the middle segment of the PCA plots next to the seed oils with a higher abundance of various chemical shifts of 0.88 ppm (acyl chain methyl protons), 1.63 ppm (methylene protons in relation to the carbonyl group, HOCO-CH_2-CH_2-), and also 2.31 and 2.33 ppm (methylene protons in relation to the carbonyl group, HOCO-CH_2-). Overall, the seed oils were grouped by the abundance of 5.33 and 5.35 ppm for proton signals of unsaturated acyl chain (Ün and Ok, 2018) and 2.05 and 2.78 ppm for the linoleic acid diallylic protons (Ingallina *et al.*, 2019). The seed oils of peanut, argan and black caraway showed higher signals of 1.31, 1.33 and 1.35 ppm for fatty acid chain methylene protons compared to the other specimens of olives, palm and coconut oils. The different abundances and shifts of several acyl chain methylene protons in the range of 1.20 to 1.3 ppm suggest different types of fatty acid chain composition for the respective samples, as postulated by Di Pietro *et al.* (2020).

The commercial palm cooking oil proton profiles showed a compact clustering for their PCA scores except for BPOM1, suggesting no significant differences between the samples despite their different packaging (bottle or pouch) and the addition of peanut and sesame oil as aroma enhancers. This result coincides with a recent assessment of commercial palm-based cooking oils sold in plastic pouches and polyethylene terephthalate (PET) bottles using gas and liquid chromatography by Hassim *et al.* (2021). A separate PCA scores plot and hierarchical clustering dendrogram for the palm cooking oils (*Figure 4*) demonstrated that apart from samples BPOM1 and BPOM4, the proton profiles of palm cooking oils of all packaging and blends are intermixed together.

A more detailed distribution of the proton profiles for all the oil specimens can be observed in a heatmap in *Figure 5* which lists the top 50 significant ^{1}H NMR signals differentiating the olive, palm, seed and coconut oils (designated as class) ranked by ANOVA. A close inspection of the heatmap revealed that the seed oils contained a higher abundance of aromatic hydrocarbons, olefins and phenolic components at region 5.00 ppm downfield (Alonso-Salces *et al.*, 2021). However, the proton signals that grouped the seed oils in the PCA were not from the downfield region as the aromatic proton signals were very low except for 5.33 and 5.35 ppm for the olefin. A magnification onto the 6.00-7.00 ppm and 3.00-4.00 ppm regions of the sesame oil ^{1}H NMR spectra to the thousandth (x1000) (*Figure 6*) as previously analysed by Schripsema (2019) and Jin *et al.* (2017) discovered the proton signals for sesamolin and sesamin lignans in very low abundance in comparison to the other proton signals. No significant signal was detected after magnification of the lignan peak regions across all the BPOM samples. However, blended cooking

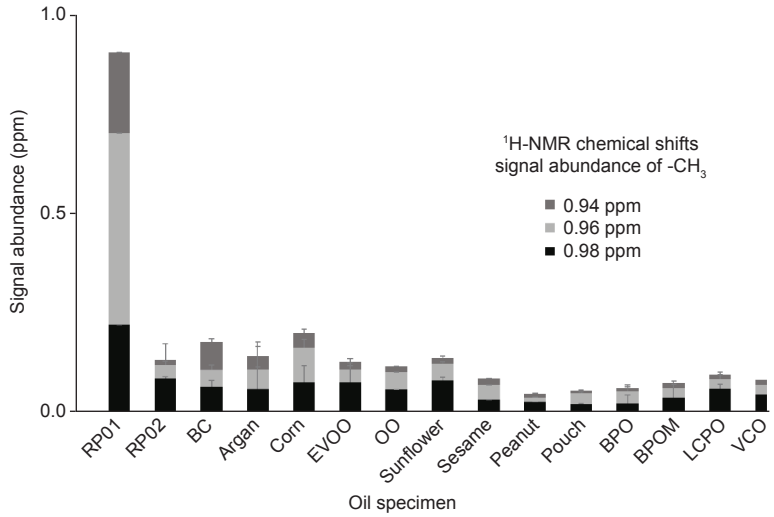

Figure 3. Average abundance of linolenic acid terminal methyl (-CH_3) group proton signals in the investigated oil specimens compared to RPO1 and RPO2.

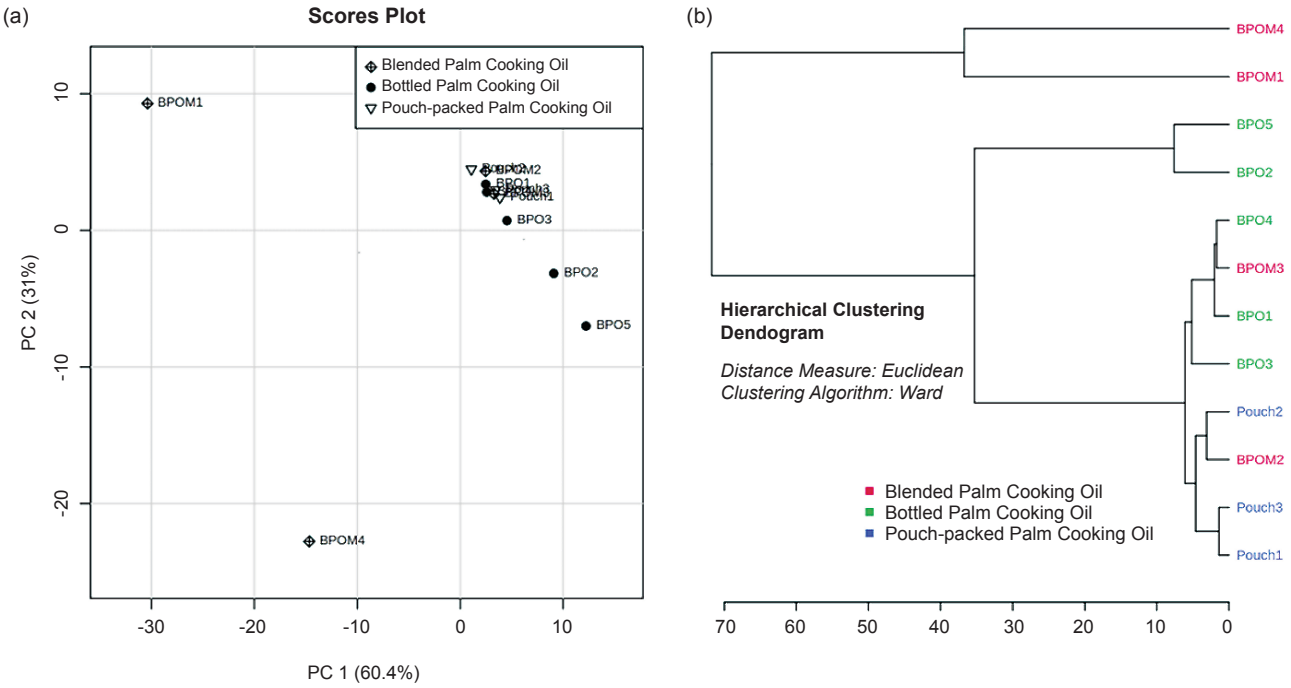

Figure 4. (a) PCA scores plot and (b) hierarchical clustering dendrogram of palm cooking oil proton NMR profiles.

oil sample BPOM4 showed similarities to the seed oil profiles in the downfield region probably due to the trace background signals from sesame and/or peanut oil. Upfield proton signal abundances at 1.35 and 2.11 ppm shifts were also detected higher for BPOM4 at levels similar to those of the peanut oil sample, suggesting comparable protons of methyl and unsaturated acyl chain (-CH_2-CH=CH).

The proton signals at 3.4 ppm observed in the seed oil and olive samples were previously reported as water presence probably due to several factors such as absorption of water during processing, *e.g.*, cooling and preservation, storage environment and hydrolytic rancidity (Cai *et al.*, 2019). It is also interesting to note that from the heatmap, the black caraway seed, olive oil, and sesame showed slight individual distinctiveness in their proton profiles while the EVOO and VCO oil samples were similarly grouped although sourced from different origins or countries.

CONCLUSION

This work demonstrates the rapid and efficient approach of ^{1}H NMR spectroscopy combined with chemometrics as a tool for the characterisation of plant oils. The different types and origins of palm oils, extra virgin and refined olive oils, virgin coconut oils, corn oils and seed oils (sesame, black caraway,

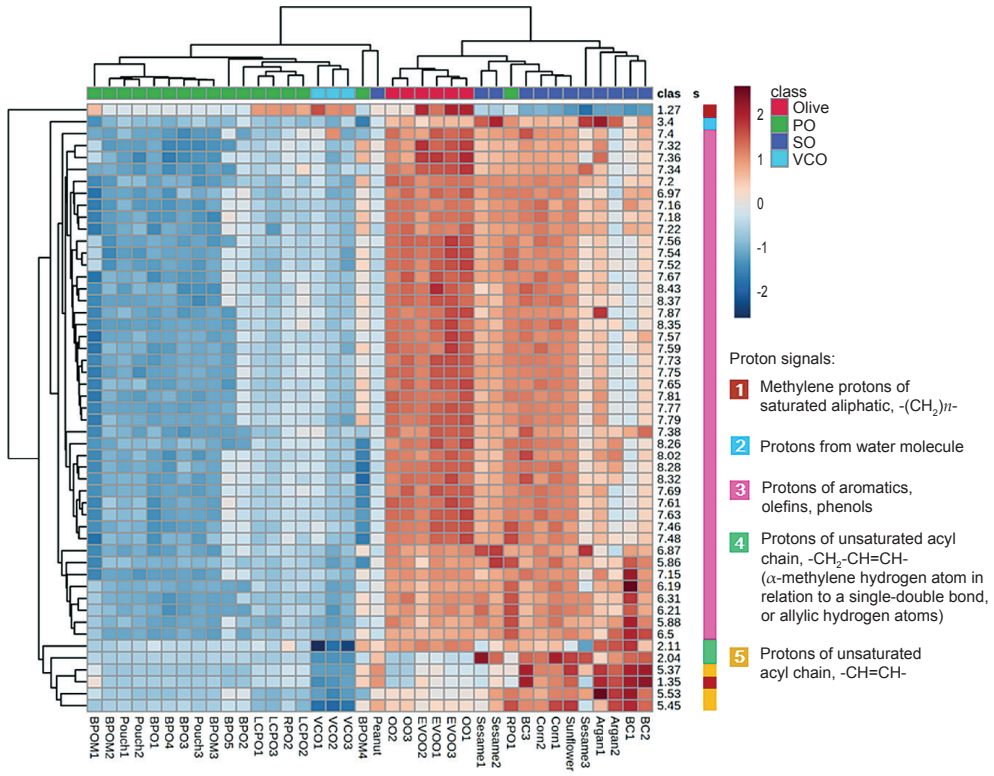

Figure 5. Heatmap of the top 50 significant plant oils proton NMR signals. Each cell corresponds to the signal abundance value from low (blue) to high (red). The signals are described according to their categories (1-5). Classes of oils: Olive and extra virgin olive oils (Olive); palm oil (PO); seed oils (SO) and virgin coconut oil (VCO).

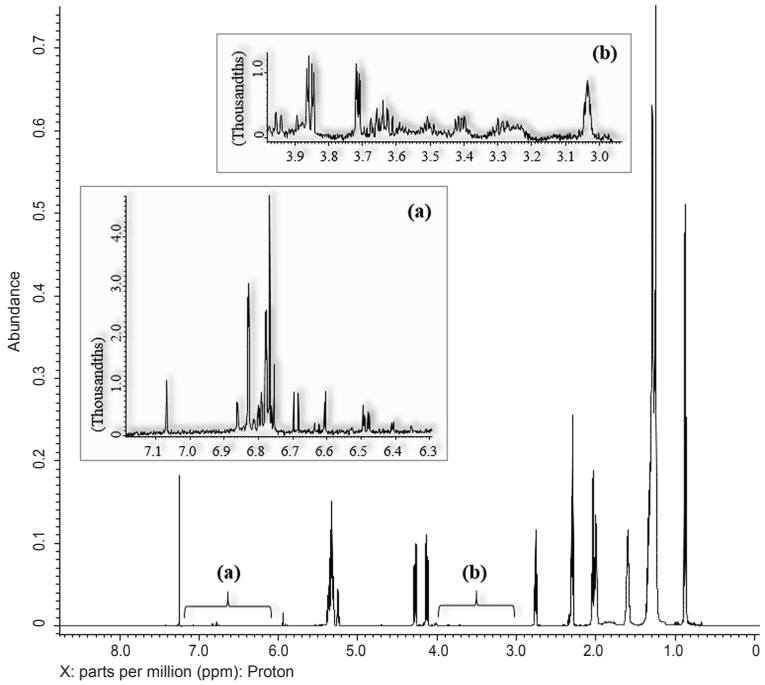

Figure 6. Magnification of the sesame oil NMR spectra to the thousandth (x1000) onto (a) 6-7 ppm and (b) 3-4 ppm proton chemical shift regions of lignans.

argan, peanut, sunflower) can be distinguished based on the similarities and differences of their proton signal abundances which correspond to their methylene (-$(CH_2)_n$-), methyl (-CH_3), unsaturated acyl chain, aromatic hydrocarbons, olefins and phenolic chemical components. The analysis requires no prior separation or treatment and the resulting spectral raw data can be processed into a statistically sound and reliable chemometric dataset to obtain maximum information. These findings can be further utilised in establishing edible oil ^{1}H NMR fingerprints for authentication and traceability purposes.

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