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ASSESSMENT ON THE CURRENT QUALITY AND IDENTITY CHARACTERISTICS OF MALAYSIAN PALM SUPEROLEIN

(Penilaian Kualiti dan Ciri Identiti Semasa Superolein Sawit Malaysia)

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Abstract

An experimental investigation was conducted to assess the current quality and identity characteristics of palm superolein (PSO) produced in Malaysia. A total of 44 PSO samples were collected from several palm oil refineries and ports in Malaysia and were tested for several important indices for characterization of PSO, *i.e.,* iodine value, fatty acids composition, triacylglycerols composition, refractive index, apparent density, relative density, slip melting point, peroxide value, impurities content, moisture content, free fatty acids content, unsaponifiable matter and saponification value. Generally, the quality of PSO was within the specification listed in Codex CXS 210-1999, with slight variations compared to the previous assessment conducted two decades ago. Most prominent variations were observed for refractive index and apparent density of PSO in which the experimental temperature was changed to 40 °C, and a new set of range values were obtained. The findings from this current quality assessment have provided significant data for the revision of the international food standard, as well as the Malaysian national standard for PSO, to accurately reflect the present quality of PSO for consumer health protection, hence removing any barriers to the international trading of PSO.

Keywords: assessment, Malaysia, palm superolein, quality, standard

Abstrak

Satu kajian eksperimen telah dijalankan untuk menilai kualiti dan ciri identiti semasa superolein sawit (PSO) yang dihasilkan di Malaysia. Sebanyak 44 sampel PSO yang diperoleh dari beberapa kilang penapisan minyak sawit dan pelabuhan di Malaysia telah diuji untuk beberapa indeks penting bagi pencirian PSO seperti nilai iodin, komposisi asid lemak, komposisi triasilgliserol, indeks biasan, ketumpatan ketara, ketumpatan relatif, takat lebur gelincir, nilai peroksida, bendasing, kelembapan, asid lemak bebas, bahan tak tersaponinan dan nilai penyabunan. Secara amnya, kualiti PSO menepati spesifikasi yang disenaraikan dalam Codex CXS 210-1999, dengan sedikit perbezaan berbanding penilaian yang telah dijalankan dua dekad yang lalu. Perbezaan ketara dilihat pada indeks biasan dan ketumpatan ketara PSO di mana suhu eksperimen telah diubah kepada 40 °C, dan satu nilai julat baru diperoleh. Hasil penemuan daripada penilaian kualiti ini telah menyediakan data yang signifikan bagi semakan semula piawaian makanan antarabangsa, dan juga piawaian kebangsaan Malaysia bagi PSO, bagi memberikan gambaran yang tepat berkenaan kualiti semasa PSO untuk penjagaan kesihatan pengguna, seterusnya menghapuskan sebarang halangan dalam perdagangan antarabangsa PSO.

Kata kunci: penilaian, Malaysia, superolein sawit, kualiti, piawai

Introduction

Oil palm (*Elaeis guineensis*) is a special crop as two types of oils are produced from the palm fruits, *i.e.*, crude palm oil (CPO) from the fleshy mesocarp, and crude palm kernel oil from the kernel [1]. Palm oil is ranked as the most consumed edible oils with 75.45 million metric tonnes of world edible oil consumption [2]. In 2020, export revenue for palm oil products increased to RM 73.25 billion due to higher export prices of palm oil [3], whereby the export volume of PSO to various destinations in the world was 67,408 tonnes worth RM 226.02 million [4]. PSO is widely consumed as cooking oil due to its high stability during frying and less tendency for cloudiness at low temperature [5].

CPO is the first product obtained during the milling of fresh fruit bunches. Native peoples in African countries consume CPO, the unrefined form of palm oil which is high in carotenes and vitamin E contents [6], albeit most people would consume it in the refined form. A further refining process turns CPO into refined palm oil, which is then subjected to fractionation to yield a more edible form of palm oil. Palm oil fractionation is an industrial process to separate liquid and solid phases of palm oil, yielding palm olein (liquid phase) and palm stearin (solid phase), with distinctive physico-chemical characteristics. Fractionation mainly involves crystallization at low temperature, and filtration to separate the two phases [7].

It has become a new interest in the market for high value added or special cuts products, *i.e.*, high iodine value (IV) olein or PSO, which is manufactured through multiple stages of fractionation as shown in Figure 1 [8]. High IV olein of 64 to 66 can even be produced through a single step fractionation, although multiple stages are often required. This special cut product is tailored to cater palm olein with higher stability towards cloudiness at low temperature settings, through the removal of saturated triacylglycerols from the olein fraction.

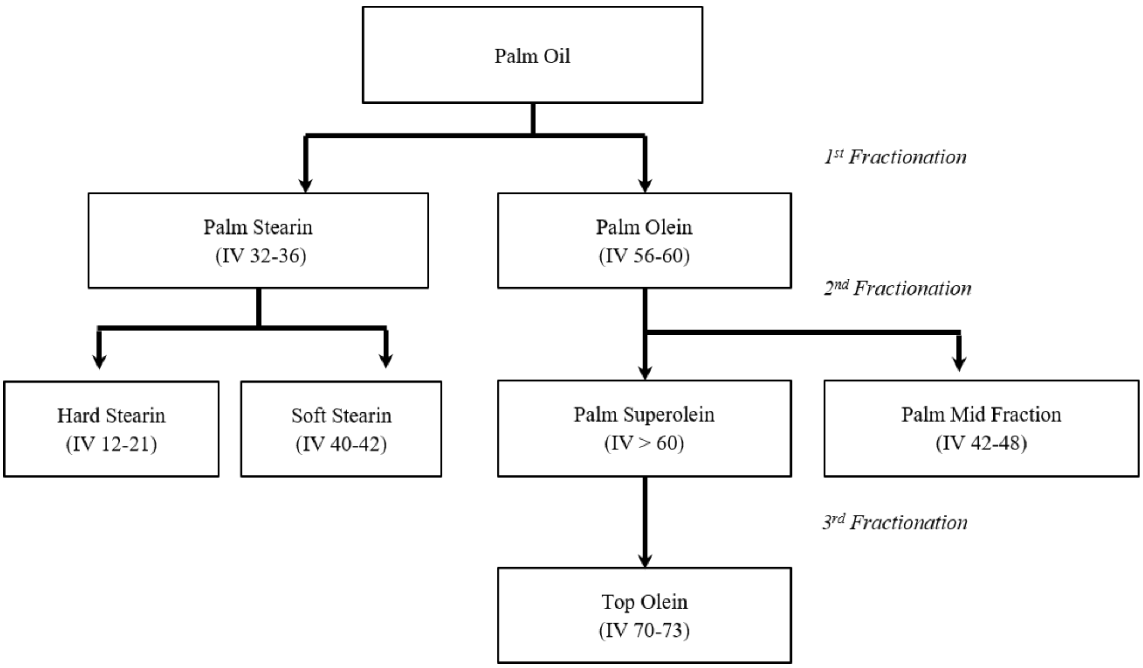

Figure 1. Multiple stages of fractionation in palm oil

PSO is the liquid fraction derived from palm oil produced through a specified controlled crystallization process to achieve a desirable IV of 60 and above [9]. It is produced by single or double fractionation. Although less demanded, it is one of the higher value fractions of palm oil. PSO has the ability to withstand colder temperatures, hence it has better resistance to clouding. It also has better clarity and stability. Generally, PSO appears transparent with light yellow in colour. Apart from being used as cooking and frying oils, it is also used for blending with polyunsaturated oils produced in temperate countries [10].

Parameters such as free fatty acids content, peroxide value and moisture content are among the important factors in determining the quality of the oil produced, and subsequently puts a price on the oil [11, 12]. Global trading of palm oil products is based on quality specifications set by international and national standards bodies. The standard specification documents commonly referred to includes Codex Alimentarius International Food Standards and Malaysian Standards. Trading specifications may also be practiced on a "willing buyer, willing seller" basis [13]. These standards are introduced to harmonise and establish uniformly accepted criteria for a certain product. The important quality parameters specified in Codex CXS 210-1999 are listed in Table 1 [9, 10].

Table 1. General characteristics of palm superolein

Parameter	**1995 Palm Superolein Assessment** [10] **(n = 32)**		**Codex CXS 210-1999** [9]
	Range	**Mean ± Standard Deviation**	
Refractive Index, RI (nD, 30 °C)	1.4631 – 1.4641	1.4634 ± 0.0003	1.459 – 1.460 (@ 40 °C)
Apparent Density, AD (g mL^{-1}, 30 °C)	0.9042 – 0.9054	0.9046 ± 0.0003	0.886 – 0.900 (@ 40 °C)
Iodine Value, IV (Wijs)	60.1 – 67.5	61.9 ± 2.15	≥ 60
Saponification Value (mg KOH g^{-1} oil)	181 -191	187 ± 3.18	180 – 205
Slip Melting Point, SMP (°C)	12.9 – 16.6	15.1 ± 0.91	-
Cloud Point (°C)	2.8 – 5.7	4.4 ± 0.71	-
Unsaponifiable Matter (%)	0.31 – 0.42	0.38 ± 0.03	≤ 13

Periodic assessments of the quality attributes of palm oil products assist in providing current information for revision of the standards, to reflect current production quality among palm oil producing companies. The previous and only published assessment on the characteristics of PSO was reported by Tang et al. [10] and presented limited information on the general characteristics and compositions of PSO (Table 1).

In view of the requirement for an up-to-date study on PSO quality, the objective of this article is to present the outcome of an assessment on the quality of Malaysian PSO for better understanding and evaluation of its current quality and identity characteristics for future improvement of the fractionation technology. The findings from this assessment were used for the revision of the Codex CXS 210-1999 Standard for Named Vegetable Oils and Malaysian Standard 1762:2004 – Palm Superolein Specifications. This round of assessment was believed to be timely as the last round of national assessment was conducted more than 25 years ago.

Materials and Methods

Sample collection

In the present study, a total of 44 refined, bleached and deodorized (RBD) PSO samples were collected from several palm oil refineries and ports in Peninsular Malaysia, Sabah and Sarawak in 2016, 2018 and 2019. One kilogram of sample was collected into a one litre opaque bottle, on a monthly basis, or whenever there was a PSO production at the refinery as PSO is usually manufactured upon request from the buyer. The samples were stored in a cool (~5 °C), dry place prior to analysis.

Chemicals and reagents

All chemical reagents and solvents used were analytical grade and high-performance liquid chromatography (HPLC) grade, and were purchased from Sigma-Aldrich, Michigan, USA.

Sample preparation

PSO samples collected were thoroughly shaken to ensure complete homogeneity according to the method described in MPOB Test Method p1.2 [14].

Physico-chemical parameters testing

The samples were subjected to physico-chemical characteristics evaluation which included iodine value, fatty acids composition, triacylglycerols composition, refractive index, apparent density, relative density, slip melting point, peroxide value, impurities content, moisture content, free fatty acids content, unsaponifiable matter and saponification value. The method of analyses was based on MPOB Test Methods, AOCS Official Methods and ISO Standard Methods. All measurements were done in triplicate, unless stated otherwise.

Free fatty acids (FFA) content

FFA content was determined according to the titration procedure based on AOCS Official Method Ca 5a-40 [15]. About 20 g of PSO sample was dissolved in neutralized *iso*-propanol and the free acids were titrated with 0.1 M sodium hydroxide solution (standardised with potassium hydrogen phthalate before usage). Phenolphthalein was added as an indicator to observe and to detect the endpoint. The endpoint is determined at the appearance of the first permanent pink colour which persists for 30 s. FFA analysis was carried out in duplicate and expressed as percentage of palmitic acid as it is the major fatty acid in palm oil.

Iodine value (IV)

The IV was analysed using the Wijs method based on the procedure described in ISO 3961 [16]. This method involved the dissolution of about 0.4 g PSO sample in a mixture of *iso*-octane and cyclohexane (1:1), and the subsequent addition of Wijs solution. The mixture was then incubated for 60 min, followed by the addition of potassium iodide (10 %) solution and 100 mL deionized water. The mixture was titrated with 0.1 M thiosulphate solution (standardised before usage) until the endpoint was reached. A blank solution was determined concurrently. Potentiometric measurement of the IV was carried out using an automated titration system T90 (Mettler Toledo, Switzerland), and the mean value of duplicate analysis was determined.

Refractive index (RI)

The RI was determined using refractometer Abbemat 300 (Anton Paar, Austria) at the temperature of 40 °C according to the method described in ISO 6320 [17]. The refractometer was calibrated daily against deionised water. A sufficient amount of PSO sample was dropped onto the measuring prism for the RI determination. The RI was determined in duplicate analysis.

Apparent density (AD)

The AD was determined at the temperature of 40 °C, using Densitometer DMA 4500M (Anton Paar, Austria), based on the method in ISO 18301 [18]. A heating element was attached to the densitometer to ensure that the samples remained melted and homogenised during the measurement. The densitometer was calibrated against air for every measurement. Duplicate analysis was carried out.

Relative density (RD)

The RD at 40 °C was determined by calculation using equation 1:

$$Relative\ density\ = \frac{Density\ of\ PSO}{0.9982} \quad (1)$$

where the density of PSO can be obtained during the measurement of apparent density and the value 0.9982 is the density of ultrapure water at 20 °C.

Moisture content

Moisture content was measured using the Karl Fischer volumetric moisture analyser (Mettler Toledo, Switzerland) according to the procedure described in ISO 8534 [19]. Samples were introduced into the flask containing Aqualine™ Complete 5 reagent (Fischer Scientific, USA). The fat content was titrated against an iodine solution and sulfur dioxide was oxidised by

iodine in the presence of water. The moisture content was measured in duplicate analysis.

Impurities content

Impurities content was determined using an oven method based on ISO 663 [20]. The sample was first filtered by using Whatman glass fibre filter paper, followed by washing with petroleum ether for several times. The filter paper with the filtrate was then heated in the oven at 103 °C for 2 h. The residue weight was measured and calculated as the impurities content in the percentage of mass fraction. The mean of duplicate analysis was reported as the impurities content.

Peroxide value (PV)

Analysis of PV was carried out using the titration technique and the potentiometric endpoint determination was based on the procedure described in ISO 27107 [21]. 5 g of PSO sample was dissolved in 50 mL acetic acid: iso-octane (3:2). Next, approximately 0.5 mL of saturated potassium iodide solution was added into the mixture and gently stirred for 1 min, followed by the addition of 30 mL deionized water. The mixture was then titrated with 0.01 M sodium thiosulphate ($Na_2S_2O_3$) solution until the endpoint was reached. The mean value of duplicate analysis was determined as the PV.

Slip melting point (SMP)

The measurement of SMP was carried out following procedures described in AOCS Official Method Cc 3-25 [22]. The sample was placed in a tube and solidified in the freezer, before heat was applied to the tube containing the sample. The sample was heated until the fat rose in the tube and the temperature at which the sample rose was recorded as the slip melting point. The measurement of SMP was carried out in triplicate.

Saponification value (SV)

The SV was analysed by titration of excess potassium hydroxide with hydrochloric acid solution, as described in ISO 3657 [23]. Approximately 2 g of sample was first mixed with 25 mL ethanolic potassium hydroxide solution and refluxed for 60 min. About 0.5 to 1.0 mL phenolphthalein indicator was added into the hot solution. The mixture was then titrated with hydrochloric acid until the pink colour of the indicator disappeared. A blank measurement was also carried out. The mean of two determinations was taken as the SV and expressed as a whole number.

Unsaponifiable matter

The unsaponifiable matter was determined following the method in ISO 3596 [24]. About 5 g of PSO was refluxed with 50 mL of potassium hydroxide solution for 1 h. The mixture was then cooled, and 100 mL of water was added and mixed well. The solution was then extracted several times with diethyl ether. Each time, the organic layer was collected and washed twice with 40 mL portions of water and once with 40 mL potassium hydroxide solution, and neutralised with 40 mL of water. The solution was evaporated at 103 °C for 15 min, allowed to cool and weighed until constant mass was obtained. The unsaponifiable matter was analysed in duplicate and the mean value was expressed as the result in percentage mass.

Fatty acids composition (FAC)

The FAC in the oil sample was measured as their corresponding methyl esters (FAME) based on the method described in ISO 12966 - Part 2 [25]. About 0.2 mL methanolic sodium methoxide was added to 0.05 g of dissolved oil sample for transmethylation to take place. After 5 min, approximately 1 mL of sodium chloride solution was added. The clear upper phase of methyl esters was carefully pipetted out and approximately 0.5 g of sodium hydrogen sulfate was added into the solution. The FAME was analysed using an Agilent GC 7890A series equipped with a flame ionization detector and a fused silica capillary column SP™ 2560 (100 m × 0.25 mm film thickness). The oven temperature was initiated at 120 °C and increased to 240 °C at 4 °C min^{-1}, then maintained for 7 min at 240 °C. Hydrogen gas was used as the carrier gas with a flow rate of 1 mL min^{-1}. The injection volume was 1 µL. The analysis was carried out in triplicate. FAC is expressed as percentage of respective fatty acids.

Triacylglycerols composition (TAG)

Quantification of TAG was carried out based on AOCS Official Method Ce 5b-89 [26] and Ce 5c-93 [27]. About 1 µL of samples dissolved in acetone was injected into

an Acquity UPLC® BEH C18 column (Waters Corp., Milford, Massachusetts, USA), with particle size of 1.7 µm, id 2.1 mm × 150 mm length, maintained at 30 °C. Isocratic elution using a mixture of acetone and acetonitrile (63.5:36.5) was carried out at a flow rate of 0.25 mL min^{-1} with a total runtime of 30 min. The detection of TAG was performed by refractive index and the TAG was quantified using the peak area normalisation method. The analysis was carried out in triplicate.

Statistical analysis

All statistical analyses were performed using Microsoft Excel 2013 (Microsoft Corporation, Redmond, WA). The linear correlation between selected quality parameters was assessed by Pearson's correlation coefficient using Minitab 19 (Minitab LLC, Pennsylvania, USA). All numerical data were expressed as the mean ± standard deviation (SD).

Results and Discussion

Physico-chemical characterisation of PSO

From the 44 samples of PSO analysed, the relevant quality control parameters such as RI, AD, IV, moisture and impurities (M&I), SMP, FFA, SV and unsaponifiable matter were found to be within the limits set by Codex CXS 210-1999 for PSO. Results are presented in Table 2 in terms of mean, standard deviation and range observed.

Table 2. Mean value with standard deviations and range of physicochemical characteristics of palm superolein

Parameter	2016/19 Palm Superolein Assessment (n = 44)			
	Mean	Standard Deviation	Min	Max
Refractive Index, RI (nD, 40 °C)	1.45933	0.00023	1.45898	1.45974
Apparent Density, AD (g mL^{-1}, 40 °C)	0.89785	0.00027	0.89720	0.89839
Iodine Value, IV (g I_2/100 g oil)	62.3	1.6	59.5	65.2
Moisture & Impurities, M&I (%)	0.05	0.01	0.02	0.08
Slip Melting Point, SMP (°C)	15.3	1.3	12.9	16.8
Free Fatty Acid, FFA (%)	0.08	0.04	0.02	0.14
Saponification Value (mg KOH g^{-1} oil)	201	10.6	181	217
Unsaponifiable Matter (%)	0.37	0.27	0.14	1.06
Peroxide Value (meq O_2 kg^{-1})	2.08	1.22	0.92	5.88

Determination of M&I contents in PSO is one of the quality requirements specified in the Malaysian Standard (MS) 1762 – Palm Superolein Specification [28]. The maximum M&I content allowed for PSO is 0.10%. The current assessment revealed that the PSO analysed had lower M&I (0.05%) than the maximum allowable limit, with a range of between 0.02% and 0.08%. Refined products should have lower amount of M&I than the crude palm oil as the crude palm oil was first subjected to drying process prior to the refining process. This step is important to avoid further hydrolysis reaction of the oil which will facilitate the increase in the FFA content of the oil [29].

FFA is commonly used as an early indicator of the quality and the acceptability of palm oil or any edible oil. It is one of the most important key quality parameters in the trading of oils and fats as it is a product of triacylglycerols hydrolytic degradation [13]. The Palm Oil Refiners Association of Malaysia (PORAM) standard specifications for RBD palm olein listed FFA as 0.1% maximum. The samples showed an average FFA content of 0.08% varying between 0.02% and 0.14%, of which were within the PORAM specifications. These ranges are acceptable as PSO is considered stable and less susceptible to hydrolytic reaction as high amount of moisture and FFA was significantly removed during the refining process [1].

PV is a parameter reflecting the degree of deterioration of palm oil products exposed to atmospheric oxygen [30]. From the PSO samples analysed, PV varied widely

from as low as 0.92 meq O_2 kg^{-1} to 5.88 meq O_2 kg^{-1}. This was probably due to the gap in time of production at the refinery as well as the time of analysis of the samples [31]. Although there was a wide variation of PV in the PSO samples, these values were acceptable by Codex which specifies PV up to 10 meq O_2 kg^{-1} for refined oils [9]. Moreover, PV is known to be unstable due to the fact that the hydroperoxides of the unsaturated fatty acids resulting from lipid oxidation are very unstable, and degrade into various volatile and non-volatile components [32]. This trend of PV instability may also be attributed to the rapid degradation of the hydroperoxides to secondary oxidation products, *i.e*., ketones, aldehydes.

RI is the measure of light ray bend angle when it goes through oil into air. It is highly dependent on the temperature it is measured, which usually is the temperature at which the oil is completely liquid [33]. It is typically used for the determination of purity together with other identity parameters. Tang et al. [10] reported that RI of PSO was in the range of 1.4631 and 1.4641, analysed at 30 °C. However, the values were not comparable to the values specified in the Codex CXS 210-1999 as the experimental temperature was not standardised to 40 °C. The recent Codex Committee of Fats and Oils in 2019 had endorsed a new range value at 40 °C. The PSO samples analysed at 40 °C had a mean value of 1.45933, with a narrow range from 1.45898 to 1.45974. This range is acceptable by Codex as it fell between the RI values of 1.459 to 1.460 as specified in Codex CXS 210-1999 [9]. RI is a physical characteristic associated to the structural property of the oil [34]. Hence, the more liquid the oil is, the higher the RI. It is important to note that the RI increases as the length of carbon chain and number of double bonds (unsaturation) of the fatty acid of the triacylglycerol increases [35], whereby the angle of refraction is reduced, and hence giving higher RI [36]. The values of RI may also be affected by oxidation and polymerisation processes [34, 37].

PSO is traded on a weight basis, but in fact measured in volume. The AD of PSO is an essential parameter for the weight determination in the cargo during shipment [10]. A similar case as in RI was observed for AD, whereby a new range value at experimental temperature of 40 °C was set at 0.886 to 0.900. In the present assessment, the AD measured at 40 °C was between 0.89720 and 0.89839 which fell within the values specified in Codex CXS 210-1999 [9]. It is important for the experimental temperature to be appropriate for testing of both RI and AD in which the oil samples are completely melted, as well as to harmonise with the reference methods recognised internationally such as ISO standard methods. The use of linear least square fit to correlate the function of RI and AD with IV, together with temperature was reported by Liew et al. [38]. Notably, both RI and AD were found to be linearly decreasing with temperature (30 to 80 °C), and linearly dependent on the IV of the oil. IV is a degree of the unsaturation of the fatty acids in the triacylglycerols. The high IV of PSO (>60) is contributed by the monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA) groups [39].

As PSO has higher IV compared to single-fractionated palm olein, it has better characteristics in terms of oil clarity, stability, and is less likely to turn cloudy [40]. Cloudiness of normal palm olein is often observed in low temperature settings during transportation and storage, as well as in cold weather [41]. This is a result of diacylglycerols (DAG) crystallisation. Although it does not affect the oil quality itself, it is always preferable by consumers to have a non-cloudy oil. Thus, the practice of blending palm olein with other soft oils, and double fractionating palm olein into PSO is applied to obtain a clearer oil. The control of the fractionation process is important to obtain a good quality product, more importantly palm olein of higher IV [42]. Optimising crystallisation temperature influences the yield and the quality of PSO. This advancement in fractionation technology has produced better quality PSO, as more solid fraction is refrained from passing through the membrane filter press, giving higher IV and improved chemical and physical properties of PSO.

PSO contains a high amount of unsaturated fatty acids (MUFA and PUFA) and the major fatty acids in PSO are mainly oleic acid (47%), palmitic acid (35%) and linoleic acid (14%) [40, 43]. Table 3 shows the FAC of PSO analysed in the current assessment. The FAC was

found to be within the range as reported in the previous articles and assessment [10, 40, 43], with oleic acid having the highest mean percentage (45.8%), followed by palmitic acid (35.7%) and linoleic acid (12.6%). The high content of unsaturated fatty acids in PSO makes it the most liquid form of palm oil products.

Table 3. Fatty acids composition of palm superolein

Fatty Acid (Weight Percent as Methyl Ester)	**2016/19 Palm Superolein Assessment (n = 44)**		**Codex CXS 210-1999** [9]
	Mean (%)	**Range (%)**	**Range (%)**
Lauric acid, C12:0	0.3	0.2 - 0.6	0.1 – 0.5
Myristic acid, C14:0	1.0	1.0 - 1.2	0.5 – 1.5
Palmitic acid, C16:0	35.7	32.5 – 38.0	30.0 – 39.0
Palmitoleic acid, C16:1	0.2	0.1 - 0.3	*ND – 0.5
Stearic acid, C18:0	3.7	3.5 - 4.2	2.8 – 4.5
Oleic acid, C18:1	45.8	43.9 – 48.7	43.0 – 49.5
Linoleic acid, C18:2	12.6	11.1 – 13.7	10.5 – 15.0
Linolenic acid, C18:3	0.3	0.2 - 0.3	0.2 – 1.0
Arachidic acid, C20:0	0.3	0.1 - 0.4	ND – 0.4

*ND: not detected

The TAG composition of PSO is listed in Table 4. In general, PSO contains mostly TAG (91%) and DAG (9%) [43]. In this study, the PSO samples analysed showed slightly higher TAG (92.51%) and lower DAG (7.49%). PSO contains more triunsaturated and monosaturated TAG, and less trisaturated TAG, hence the greater IV. Additionally, the much lower trisaturated TAG content in PSO contributed to its resistance towards cloudiness. With regards to the correlation of TAG composition with IV, the function of TAG composition by HPLC analysis to predict the IV of the oil was reported by Haryati et al. [44]. The IV was estimated from the regression model mainly on the peak areas of the disaturated, trisaturated and triunsaturated TAGs.

The SMP is the temperature at which a fat change from solid to liquid phase. It is dependent on the FAC of the oil. The average SMP observed from the PSO analysed was 15.3 °C, with range values between 12.9 °C and 16.9 °C. These values were similar to those reported by Tang et al. [10], and they also reported that PSO with high IV had lower SMP as a result of complex composition of the PSO itself which is a mixture of various species of acylglycerols.

The SV is used to measure the types of acylglycerols in the oil by measuring alkali-reactive groups in the oil. A higher SV indicates low-molecular weight triacylglycerols of edible fats and oils. The range observed for SV was 181 to 217 mg KOH g^{-1}, a slightly wider range compared to the previous assessment in 1995. This range conformed to the lower limit of SV (180 mg KOH g^{-1}) in Codex CXS 210, and slightly exceeded the upper limit of 205 mg KOH g^{-1}. Low SV for PSO is attributable to the high amount of long chain fatty acids, *i.e.* oleic and linoleic acids in the composition of PSO.

The unsaponifiable matter is the fraction of substances in the PSO after saponification, which is not volatile at 103 °C that includes sterols, higher hydrocarbons and alcohols, aliphatic and terpenic alcohols, and foreign matter extracted by diethyl ether [45]. An average value of 0.37% of unsaponifiable matter was observed for PSO. Palm oil is classified as a vegetable oil having low unsaponifiable matter of less than 1.2% [46]. The previous study [46] also stated that sterols are the predominant unsaponifiable matter in most oils. In palm oil, Codex CXS 210-1999 listed β-sitosterol, campesterol and stigmasterol as the three main sterols found in PSO. Tocopherols and tocotrienols fractions are also abundant in palm oil products, in the range of 400 to 1400 mg kg^{-1} [9]. These minor components are highly useful in food applications as well as play a significant role in both human and animal nutrition.

Table 4. Acylglycerol composition of palm superolein

	2016/19 Palm Superolein Assessment (n = 44)	
Glycerol	**Mean (%)**	**Range (%)**
DAG	7.49	6.20 – 10.44
Triunsaturated TAG		
*OLL	0.63	0.52 – 0.88
OLO	2.38	2.04 – 2.67
OOO	5.36	4.55 – 6.37
Monosaturated TAG		
PLL	3.27	2.66 – 3.68
PLO	12.40	10.84 – 13.94
POO	31.18	28.73 – 35.25
SOO	3.12	2.71 – 3.55
Disaturated TAG		
MLP	0.71	0.61 – 0.79
PLP	11.22	10.13 – 12.14
POP	18.30	12.54 – 22.99
POS	3.31	2.42 – 4.11
SOS	0.43	0.29 – 0.56
Trisaturated TAG		
MPP	0.10	0.03 – 0.93
PPP	0.13	0.06 – 0.51

*O – oleic; L – linoleic; P – palmitic; S – stearic; M – myristic

Correlation analysis of selected quality and identity parameters

The linear relationship for IV, RI, AD, FFA and moisture of the PSO samples was assessed using Pearson correlation. The correlation results shown in Table 5 demonstrates a positive relationship for RI-IV, AD-IV and FFA-moisture. A very strong positive correlation was observed for AD-IV with *p*-value of 0.000 (p <0.05), indicating the highly significant correlations. A similar observation was noted for FFA-moisture and this is expected as FFA is highly influenced by moisture content in the oil. Correlations between RI-AD and RI-IV were somewhat insignificant (p >0.05). More data are required to confirm and to derive conclusive correlations between each parameter.

Table 5. Correlation of selected parameters in PSO quality performance

Parameter	**Pearson Correlation Coefficient**	***p*-value**
RI-AD	-0.011	0.944
RI-IV	0.012	0.939
AD-IV	0.843	0.000
FFA-Moisture	0.538	0.012

Conclusion

The present study concludes that the physical characteristics and the chemical compositions of PSO have experienced slight variations over the last two decades, particularly in its RI and AD. This shift suggests that the current advancement in the

fractionation technology has produced better quality PSO for its various uses in the food industry. The overall quality performance of PSO was satisfactory and largely conformed to Codex CXS 210-1999. Periodic evaluation of PSO has helped in understanding the current situation of PSO produced in Malaysia in terms of its quality and the setting of realistic and achievable targets in the quality specification. These new sets of quality data will be used in the revision of guidelines for the identity characteristics of PSO in MS 1762:2004, Palm Superolein - Specification.

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References

1. Tan, C. H., Ghazali, H. M., Ainie, K., Tan, C. P. and Ariffin, A. A. (2009). Extraction and physicochemical properties of low free fatty acid crude palm oil. *Food Chemistry*, 113: 645-650.
2. Statista (2020). Consumption of vegetable oils worldwide from 2013/14 to 2021/2022, by oil type. https://www.statista.com/statistics/263937/vegetable-oils-global-consumption/ [Access online 15 September 2021].
3. Parveez, G. K. A., Tarmizi, A. H. A., Sundram, S., Loh, S. K., Ong-Abdullah, M., Kosheela Devi, P. P., Mohamed Saleh, K., Mohd Ishak, S. and Zainab, I. (2021). Oil palm economic performance in Malaysia and R&D progress in 2020. *Journal of Oil Palm Research*, 33(2): 181-214.
4. Malaysian Palm Oil Board (MPOB) (2020). Malaysian Oil Palm Statistics 2020. MPOB, Malaysia: pp. 75.
5. Pande, G., Akoh, C. C. and Lai, O. M. (2012). Palm Oil: Production, Processing, Characterization and Uses. AOCS Press, USA: pp. 576-577.
6. Palm Oil World (2020). Nutrition benefits. http://www.palmoilworld.org/nutrition.html [Access online 23 November 2020].
7. Gee, P.T. (2007). Analytical characteristics of crude and refined palm oil and fractions. *European Journal of Lipid Science and Technology*, 109: 373-379.
8. Gibon, V. (2012). Palm oil: Production, processing, characterization and uses. AOCS Press, USA: pp. 364-365.
9. Food and Agriculture Organization of the United Nations and World Health Organization. Codex Alimentarius International Food Standards - CODEX Standard for Named Vegetable Oils Codex CXS 210-1999 [Access online 15 September 2021].
10. Tang, T. S., Chong, C. L., Yusoff, M. S. A. and Ab Gapor, M. T. (1995). Characteristics of superolein from the fractionation of palm oil. *PORIM Technology*, 17: 1-9.
11. Endo, Y. (2018). Analytical methods to evaluate the quality of edible fats and oils: The JOCS standard methods for analysis of fats, oils and related materials (2013) and advanced methods. *Journal of Oleo Science*, 67(1): 1-10.
12. Decker, E., Elias, R., McClements, J.D. (2010). Oxidation in foods and beverages and antioxidant applications. Elsevier, Amsterdam: pp. 432.
13. Chong, C. L. (2012). Palm Oil: Production, Processing, Characterization and Uses. AOCS Press, USA: pp. 445.
14. Malaysian Palm Oil Board (2004). MPOB Test Methods: A compendium of test on palm oil products, palm kernel products, fatty acids, food related products and others. MPOB, Malaysia: pp. 153-155.
15. American Oil Chemists' Society (AOCS) (2017). Official methods and recommended practices of the AOCS, 7th ed. AOCS Press, USA: Method Ca 5a-40.
16. International Organization for Standardization (ISO) (2018). ISO 3961. Animal and vegetable fats and oils - Determination of iodine value. ISO, Switzerland.
17. International Organization for Standardization (ISO) (2017). ISO 6320. Animal and vegetable fats and oils - Determination of refractive index. ISO, Switzerland

18. International Organization for Standardization (ISO) (2014). ISO 18301. Animal and vegetable fats and oils - Determination of conventional mass per volume (litre weight in air) - Oscillating U-tube method. ISO, Switzerland.
19. International Organization for Standardization (ISO) (2017). ISO 8534. Animal and vegetable fats and oils - Determination of water content - Karl Fischer method (pyridine free). ISO, Switzerland.
20. International Organization for Standardization (ISO) (2017). ISO 663. Animal and vegetable fats and oils - Determination of insoluble impurities content. ISO, Switzerland.
21. International Organization for Standardization (ISO) (2008). ISO 27107. Animal and vegetable fats and oils - Determination of peroxide value - Potentiometric end-point determination. ISO, Switzerland.
22. American Oil Chemists' Society (AOCS) (2017). Official methods and recommended practices of the AOCS, 7th ed. AOCS Press, USA: Method Cc 3-25.
23. International Organization for Standardization (ISO) (2020). ISO 3657. Animal and vegetable fats and oils - Determination of saponification value. ISO, Switzerland.
24. International Organization for Standardization (ISO) (2000). ISO 3596. Animal and vegetable fats and oils - Determination of unsaponifiable matter - Method using diethyl ether extraction. ISO, Switzerland.
25. International Organization for Standardization (ISO) (2017). ISO 12966-2. Animal and vegetable fats and oils - Gas chromatography of fatty acid methyl esters - Part 2: Preparation of methyl esters of fatty acids. ISO, Switzerland.
26. American Oil Chemists' Society (AOCS) (2017). Official methods and recommended practices of the AOCS, 7th ed. AOCS Press, USA: Method Ce 5b-89.
27. American Oil Chemists' Society (AOCS) (2017). Official methods and recommended practices of the AOCS, 7th ed. AOCS Press, USA: Method Ce 5c-93.
28. Malaysian Standard (2004). MS 1762. Palm Superolein – Specification. ICS: 67.200.10. Department of Standards, Malaysia.
29. Sherine, S. M. A.E. and Basma, M. M. E. (2020). Precision Agriculture Technologies for Food Security and Sustainability. 1st Ed. USA, IGI Global: pp. 437.
30. Frankel, E. N. (2012). Lipid Oxidation. Woodhead Publishing Limited, USA: pp. 104.
31. Almeida, D. T., Viana. T. V., Costa, M. M., Silv,a C. S. and Feitosa, S. (2019). Effects of different storage conditions on the oxidative stability of crude and refined palm oil, olein and stearin (*Elaeis guineensis*). *Food Science and Technology,* 39(1): 211-217.
32. Kaleem, A., Aziz, S., Iqtedar, M., Abdullah, R., Aftab, M., Rashid, R., Shakoori, F. R. and Naz, S. (2015). Investigating changes and effect of peroxide values in cooking oils subject to light and heat. *FUUAST Journal of Biology,* 5(2): 191-196.
33. Hishamuddin, E., Sulaiman, N., Ahmad Bustamam, F. K. and Yeoh, C. B. (2020). Recent updates on the Codex standard for named vegetable oils (CXS 210-1999) in relation to palm oil and palm kernel oil. *Palm Oil Developments,* 72: 34-41.
34. Godswill, A. C., Omagwula, I. O., Victory, I. S. and Gonzaga, A. I. (2018). Effects of repeated deep-frying on refractive index and peroxide value of selected vegetable oils. *International Journal of Advanced Academic Research,* 4 (4): 106-119.
35. Al Majidi, M. I. H. and Bader, A. T. (2015). Physicochemical characteristics of some imported edible vegetable oils in Iraq. *Research Journal of Pharmaceutical, Biological and Chemical Science, 6(5): 488-494.*
36. Imoisi, O. B., Ukhun, M. E., Ezoguan, V. O. and Osemwegie, Q. E. (2018). Fatty acid profiles, correlation of iodine value, refractive index of heated, unheated palm kernel oil and palm olein. *Journal of Chemical Society of Nigeria*, 43 (4): 745-751.
37. Patterson, H. B. W. (2009). Bleaching and Purifying Fats and Oils: Theory and Practice. AOCS Press, USA: pp. 18.

38. Liew, K. Y., Latiff, N. A., Nordin, M. R. and Seng, C. E. (1995). Densities and refractive indices of hydrogenated palm olein and fractionated palm oil. *Elaeis*, 7(2): 159-164.
39. Hashem, H. A., Nasser, E. A., Ghareeb, A. A. and Adel, G. A. (2018). Industrial scale production of palm super olein using modified and innovative dry fractionation technique. *Egyptian Journal of Chemistry*, 61: 1-11.
40. Malaysian Palm Oil Board (MPOB) (2009). Pocketbook of Palm Oil Uses. MPOB, Malaysia: pp. 23.
41. Idris, N. A., Jamaludin, R. and Hassan, H. (2003). Process to prevent and delay clouding in palm olein. *US Patent* No. 20030068426 A1.
42. Che Man, Y. B., Haryati, T., Ghazali, H. M. and Asbi, B. A. (1999). Composition and thermal profile of crude palm oil and its products. *Journal of the American Oil Chemists' Society,* 76(2): 237-242.
43. Goon, D. E., Siti Hamimah, S. A. K., Ab Latip, N., Ab Rahim, S. and Mazlan, M. (2019). Palm oil in lipid-based formulations and drug delivery systems. *Biomolecules,* 9(2): 64.
44. Haryati, T., Che Man, Y. B., Ghazali, H. M., Asbi, B. A. and Buana, L. (1998). Determination of iodine value of palm oil based on triglyceride composition. *Journal of the American Oil Chemists' Society,* 75(7): 789-792.
45. Malaysian Palm Oil Board (2004). MPOB test methods: A compendium of test on palm oil products, palm kernel products, fatty acids, food related products and others. MPOB, Malaysia: pp. 198-203.
46. Rao, B. S. N. (2001). Nonglyceride components of edible oils and fats. 1. Chemistry and distribution. *Food and Nutrition Bulletin*, 22(1): 81-86.