

THE EFFECTS OF RECYCLING PALM PRESSED-FIBRE OIL ON CRUDE PALM OIL QUALITY

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ABSTRACT

Palm pressed-fibre oil (PPFO) is known as a phytonutrients rich oil. PPFO was practically recycled back to the crude palm oil (CPO) processing line to increase oil extraction rates. This study evaluated the effect of blending CPO with PPFO at different PPFO dosage from 5%-25% (w/w) on blended oil quality. Free fatty acid (FFA) of blended oils increased from 3.42%-3.91%, while deterioration of bleachability index (DOBI) and iodine value (IV) decreased from 2.76 to 2.33 and 51.96 ± 0.37 to 50.21 ± 0.03 , respectively. Minor components amount in blended oils were found to increase with carotenoids (539-654 ppm), squalene (475-1201 ppm), sterols (433-808 ppm), phospholipids (34.67-63.84 ppm) and vitamin E (1185-1626 ppm). The oxidative stability of blended oils was improved (14.31-16.76 hr) but chlorine, iron and copper content were found to increase from 3.85-7.49 ppm, 13.58-17.63 ppm and 0.19-0.38 ppm, respectively. Although blending PPFO would deteriorate some quality properties of CPO, nonetheless, PPFO is rich with phytonutrients, therefore signifying its potential applications in pharmaceuticals and food industry.

Keywords: blending PPFO, chloride, crude palm oil, heavy metals, oil quality, palm pressed-fibre oil.

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INTRODUCTION

The rapid increase in palm oil production over the last few decades has made it as one of the most important oils in the world. Malaysia contributes to almost 30% of global palm oil production and 36% of the world exporters in 2019 (Workman, 2020). Parveez *et al.* (2020) reported that oil palm planted areas in Malaysia increased to 5.90 million hectares and fresh fruit bunches (FFB) yield of 17.19 t ha⁻¹ in 2019. This would contribute to a generation of massive amount of solid wastes, in the form of lignocellulosic residues from replanting activities and oil mill processing, stemming from oil palm trunk (OPT), oil palm frond (OPF), empty fruit bunches (EFB), palm pressed mesocarp fibre (PPMF), palm kernel cake (PKC), palm kernel shell (PKS) and decanter cake. Solid wastes produced

were 20%-28% EFB, 11%-12% PPMF and 5%-8% PKS (Awalludin *et al.*, 2015; Onoja *et al.*, 2019; Sarmidi *et al.*, 2009; Yusoff, 2006).

PPMF is a by-product produced after crude palm oil (CPO) is extracted from the digested oil palm fruits mash, using a screw press. PPMF contains a substantial amount of residual oil (5%-8%) that is usually burnt along with the PPMF in the boiler, in order to supply energy to the mill. Several techniques to recover the residual oils from PPMF have been reported, such as, solvent extraction, enzymatic treatment, supercritical carbon dioxide (SC-CO₂) and remnant oil recovery (ROR) system (Lau *et al.*, 2006; Noorshamsiana *et al.*, 2017; Nur Sulihatimarsyila *et al.*, 2019; Vijaya *et al.*, 2013). The residual oil extracted from this technique is known as palm pressed-fibre oil (PPFO). The commercial extraction technique of PPFO from PPMF is solvent extraction using hexane (Nur Sulihatimarsyila *et al.*, 2019). According to a survey done by Malaysian Palm Oil Board (MPOB), the system has been commercially installed in 12 palm oil mills in Malaysia (Umami Kalsum and Rohaya, 2019).

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Non-polar solvent, namely hexane is utilised for extraction. This technique has been used in various vegetable oil extractions, such as rapeseed and soybean (Kumar *et al.*, 2017). Many studies reported that PPFO is rich with carotenoids, vitamin E (tocopherol and tocotrienols) (Choo *et al.*, 1996; Goh *et al.*, 1985; Lau *et al.*, 2017; Ng and Choo, 2011; Noorshamsiana *et al.*, 2017; Nur Sulihatimarsyila *et al.*, 2019; Rusnani *et al.*, 2012), squalene and sterols (Choo *et al.*, 1996; Goh *et al.*, 1985; Lau *et al.*, 2007; Ng and Choo, 2011; Phoon *et al.*, 2018). Since PPFO is rich in natural phytonutrients, almost all previous studies had suggested that PPFO has a wide range of applications in pharmaceuticals, supplementation and cosmetics (Lau *et al.*, 2017; Nur Sulihatimarsyila *et al.*, 2019). PPFO was also found to contain high amounts of water-soluble compounds, such as phospholipids (217-1063 ppm) and phenolics with outstanding antioxidant activities (Lau *et al.*, 2007; Ummi Kalsum and Rohaya, 2019; Valeria *et al.*, 2017). Apart from the normal fatty acid composition (FAC) content, PPFO has high diacylglycerol (DAG) and triacylglycerol (TAG) ($67.04\% \pm 0.05$) with acceptable amount of monoacylglycerol (MAG)/ free fatty acid (FFA) ($13.02\% \pm 0.02$) compositions. Nonetheless, PPFO also contains substantial amount of lauric acid (5.89%-9.09%) due to broken kernels in the mesocarp fibre, thus, making it suitable for the food applications and industry (Lau *et al.*, 2006; Neoh *et al.*, 2011).

However, studies also showed that PPFO is low in quality compared to CPO. Ummi and Rohaya (2019) reported on the deterioration of bleachability index (DOBI) of the PPFO, which ranges from 1.15-2.17 while the FFA ranges from 5.09%-10.6% as shown in Table 1. Also, PPFO has a high content of chlorine (207.62-379.58 ppm) which will lead to formation of carcinogenic compounds such as 3-monochloropropane-1,2-diols (3-MCPD) esters (Table 1) (Ramli *et al.*, 2015; Ummi Kalsum and Rohaya, 2019). Therefore, PPFO can only be sold at a lower price as compared to CPO because PPFO has not been classified into any oil classification set by MPOB. Since it is chemically extracted, it is therefore forbidden to blend PPFO into the CPO processing line (Ummi Kalsum and Rohaya, 2019). Since PPFO is not under any of the palm oil categories, it is automatically classified as sludge oil, which is the lowest quality of palm oil grade, in terms of price. As a result, palm oil millers tend to blend PPFO into CPO to increase its oil extraction rate. Apparently, the practice of blending PPFO into CPO has been increasing in recent years, as indicated by the increment of phosphorous content and FFA in CPO (Ummi Kalsum and Rohaya, 2019). According to the Code of Good Milling Practice of Palm Oil Mills (GMP), residual oils cannot be recycled or blended with CPO,

mainly due to quality deterioration. Nevertheless, the blending effect of PPFO on CPO quality is not extensively studied and reported. Therefore, this study is aimed to examine the quality of PPFO from commercial solvent extraction plant as well as to evaluate the characteristics of blended oil when different dosage of PPFO is used to blend with CPO.

MATERIALS AND METHODS

Materials

CPO and solvent extracted PPFO were collected from eight palm oil mills and eight solvent extraction plants located in central, southern and northern region of Peninsular Malaysia. The samples were freshly collected and kept in the chiller at 4°C prior to the blending study. The PPFO samples collected from eight solvent extractions were used to determine the initial quality properties of PPFO. While, PPFO and CPO samples collected from Sitiawan, Perak and Labu Palm Oil Mill, Negeri Sembilan, Malaysia respectively, were used to evaluate the effect of blending CPO with PPFO at different dosages from 0%-25% (w/w) at 60°C and 300 rpm for 20 min on residual hexane concentration, FFA, DOBI, oxidative stability, iodine value (IV), trace elements, chloride content, phytonutrients content, FAC and acylglycerols compositions. All solvents, chemicals and reference standards (analytical grades) used in this study were purchased from Merck, JT Baker and Sigma, Germany.

Analysis of Quality Properties

The PPFO, CPO and blended oils samples were analysed according to MPOB Test Method (MPOB, 2005) to quantify the FFA, DOBI, carotene content, IV and phosphorus content.

Hexane Residual Analysis using Headspace Chromatography

Headspace analyses was performed by using GC-FID (6890N; Agilent Technologies, Santa Clara, CA, USA) and the headspace-sampler (HS-20; Shimadzu), which was used as preprocessing device (Jeong *et al.*, 2017; Kumar and Gow, 1994). Gas flow for analysis was 1.0 mL min^{-1} , and the detector and injection temperatures were regulated at 100°C and 50°C, respectively. The column used was $30 \text{ m} \times 0.3 \text{ mm} \times 0.2 \text{ }\mu\text{m}$ coated with methylpolysiloxane. The analysis was implemented in a 100:1 split mode by applying helium as carrier gas. Residual solvent content was expressed in mg kg^{-1} (ppm) of the sample.

Heavy Metals Concentration Analysis using Flame Atomic Adsorption Spectroscopy (FAAS)

The metals content such as iron (Fe) and copper (Cu) was determined using Atomic Adsorption Spectrophotometer (AA-7000F, Shimadzu, Japan). Samples of 2 g each were weighed in 250 mL round bottom flask, later added with 5 mL of nitric acid (HNO₃), 5 mL sulphuric acid (H₂SO₄) and 2 mL hydrogen peroxide (H₂O₂). The mixture was then heated using water bath at 100°C until a clear solution was obtained. The solution was then filtered and made up to 50 mL using ultrapure water and transferred to a plastic bottle for metal analysis by atomic adsorption spectroscopy (AAS).

Total Chlorine Content Analysis

The amount of chloride in CPO, PPFO and blended oils was measured using a Total Chlorine Analyser (TCA) NSX-2100H (Mitsubishi Chemical Analytech, Kanagawa, Japan). Samples were prepared according to American Society for Testing and Materials (ASTM) D4929-04 method (ASTM, 2004). The operation principle of TCA was combustion and microcoulometry. In the presence of oxygen and argon as a carrier gas, 0.065 g of oil aliquot was combusted in the TCA at temperatures between 800°C-1000°C. The hydrogen chloride released as a result of the combustion was then titrated with silver ion emitted from the titration cell. Chlorine content is quantified based on the intensity of electrical current released in the microcoulometer throughout the titration stage (Azmil *et al.*, 2019).

Oxidative Stability Analysis

The oil stability was determined using Rancimat Analyser (743 Metrohm, Switzerland). Oil samples of 3.0 ± 0.1 g each were weighed in a glass vessel. The temperature of heating block was set to 120°C, while the gas flow was set to 20 L hr⁻¹ (Farhoosh, 2007).

Fatty Acid Compositions (FAC) Analysis

FAC were determined using gas chromatography flame ionisation (GC-FID) (Agilent Technologies). The samples were analysed according to American Oil Chemists' Society (AOCS) Official Method Ce 2-66 (AOCS 1997).

Triacylglycerols (TAG) Analysis by Ultra-high-Performance Liquid Chromatography (U-HPLC)

TAG analysis was performed by U-HPLC (ACQUITY UPLC H-Class System, Waters Corp., Milford, Massachusetts, USA) according to the AOCS Official Methods Ce 5c-89 and Ce 5c-93 (AOCS 1997). Individual samples were solubilised in acetone to form a 5% w/v solution. The solution was then filtered through a 0.2 µm syringe filter. Exactly 1 µL of the solution was then injected through an ACQUITY UPLC® BEH C18 column (Waters Corp., Milford, Massachusetts, USA) with particle size of 1.7 µm, i.d. 2.1 mm × 150 mm length, and maintained at 30°C. The flowrate was fixed at 0.25 mL min⁻¹. TAG were detected using refractive index detector set at 35°C with full scale setup at 100 µRIU for 20-30 min of total run times.

TABLE 1. INITIAL QUALITY OF CRUDE PALM OIL (CPO) AND SOLVENT EXTRACTED PALM PRESSED-FIBRE OIL (PPFO) FROM DIFFERENT COMMERCIAL PLANTS

	CPO (this study)	PPFO commercial I (this study)	PPFO commercial II (Ummi Kalsum and Rohaya, 2019)	PPFO commercial III (Nur Sulihatimarsyila <i>et al.</i> , 2019)	MS 814:2007 specification (Farah Khuwailah <i>et al.</i> , 2019)
FFA (%)	3.1 – 4.5	5.38 – 8.26	5.09 – 10.6	5.30 ± 0.19	<5%
DOBI value	2.43 – 2.83	1.62 – 2.02	1.15 – 2.17	2.02 ± 0.18	>2.3
Oxidative stability (hr)	14 – 17	18 – 25	-	-	-
Iodine value (ppm)	49.11 – 51.22	51.96 – 53.22	-	-	52 ± 0.66
Phosphorus content (ppm)	16 – 27	214 – 247	217 – 1 063	633 ± 83	<10
Chloride content (ppm)	1.95 – 10.89	23.41 – 127.23	207.62 – 379.58	-	-
Carotene content (ppm)	537 – 572	1 258 – 1 750	1 025 – 1 913	1 467 ± 35	500 – 600
Vitamin E (ppm)	771 – 1 108	1 686 – 2 600	1 574 – 2 999	1 527 ± 73	-
Hexane concentrations (ppm)	N/A	111 – 4 046	-	-	-

Note: The solvent extracted PPFO was collected from: (I) eight, (II) nine, (III) one, solvent extraction plants in Peninsular Malaysia.

Results are mean values and ± standard deviation (n=3) of oil samples.

FFA – free fatty acid; DOBI - deterioration of bleaching index; N/A - not applicable.

Vitamin E

The vitamin E (tocopherols and tocotrienols) content was determined using HPLC and fluorescence detector (Agilent Technologies), with C18 column (150 mm x 4.6 mm i.d.). Mobile phase was acetonitrile/methanol (50:50 v/v) with 1.0 mL min⁻¹ flow.

Squalene and Sterols

CPO, PPFO and blended samples were pre-treated using saponification method prior to analyses of sterols and squalene following the MPOB Test Method (MPOB, 2005). Samples weighing 5.0 g each were mixed with 2.5 g 10% (w/v) potassium hydroxide (KOH) in an ethanolic solution. The mixture was refluxed for 1 hr at temperatures between 70°C-80°C. Hexane was used to extract the unsaponifiable matters from the saponified mixture. Hexane was then removed from the mixture using rotary evaporator and the extracts were then washed using distilled water until a neutral pH was obtained. The remaining moisture was removed using sodium sulphate (Na₂SO₄). The samples were then dissolved in 100% *n*-hexane to an appropriate concentration. The diluted samples were analysed using gas chromatography (GC) Autosystem XL, Perkin Elmer with FID. The samples were separated in the capillary column purchased from Supelco SACTM -5 (Sigma) (30 mm x 0.25 mm i.d.) bonded with a 0.25 µm film of 5% phenyls/95% dimethylpolysiloxane in the presence of helium gas as mobile phase (Nor Faizah *et al.*, 2020).

RESULTS AND DISCUSSION

Effect on Residual Hexane Concentration

Figure 1 shows the typical flow process of the solvent extraction system that has been commercially used in Malaysia. A non-polar solvent, hexane, is utilised for extraction. This solvent has been used in various vegetable oil extractions, such as rapeseed and soybean (Kumar *et al.*, 2017). Hexane is one of the most widely used solvents in edible oil extractions due to its excellent performance in oil recovery, high solubilising ability and evaporation at narrow boiling temperature (63°C-69°C) (Kumar *et al.*, 2017). Solvent extraction for PPFO production uses high temperatures between 80°C-140°C. The solvent, evaporated through desolventisation of PPF in the desolventiser-toaster (Figure 1) and from miscella in the distillation column was carried over into atmospheric condenser to be converted into liquid and later deposited back into the solvent tank (Serrato, 1981). However, there is still excess solvent that would probably be trapped in the miscella which

contains oil, gums, waxes and water. Hexane would likely be eliminated further during the refining stage since it is a compulsory process before the oil can be used as a food product. The final product could still contain traces amount of hexane. Several studies revealed that hexane is the most toxic of the alkanes group. If ingested, it causes nausea, vertigo, bronchial and intestinal irritation and affects the central nervous system. A study showed that there was no effect on human if exposed at concentration of up to 2000 ppm. However, it would cause dizziness, slight nausea, headache, and eye and throat irritations if exposed at 5000 ppm concentration (Clough, 2014). Therefore, it is desirable for the trace hexane in edible oil to be as low as possible.

Maximum residual level (MRL) of hexane has been established by various organisations and countries. European Union (EU) regulates the limit of hexane residues at 1 ppm (maximum) (Yousefi and Hosseini, 2017), while in Korea, the maximum level is 5 ppm (Oh *et al.*, 2005). Static headspace analyses using GC-FID and gas chromatography mass spectrometric detector (GC-MSD) were developed to detect residual hexane in food and edible oil products (Jeong *et al.*, 2017; Oh *et al.*, 2005; Othman *et al.*, 2019; Yousefi and Hosseini, 2017). Table 1 shows the residual hexane concentrations in PPFO collected from eight solvent extraction plants in Malaysia. The hexane's concentration in PPFO ranged from 111-4046 ppm. Results showed that the hexane level in blended oils increased with the increment in PPFO dosage (from 5%-25% w/w of CPO), ranging from 3 ± 0.04 ppm to 18 ± 0.2 ppm, respectively (Figure 2). These results indicated that hexane level in blended oils depended on the initial concentration of residual hexane in PPFO.

Effect on Free Fatty Acid (FFA)

The FFA content in palm oil indicates the deterioration level of oil, which then determines the value of the oil (Japir *et al.*, 2017; Saad *et al.*, 2007). Table 1 depicts the respective quality of CPO and PPFO from this study compared to other researchers' findings. Results showed that PPFO has higher FFA values compared to CPO. The FFA values of PPFO and CPO were 5.38%-8.26% and 3.1%-4.5%, respectively. The maximum level of FFA value in CPO is below 5%, as specified by Palm Oil Refiners Association Malaysia (PORAM) in Malaysian Standard MS814:2007 (Farah Khuwailah *et al.*, 2019). Umami Kalsum and Rohaya (2019) reported that FFA values of PPFO produced from nine solvent extraction plants in their survey were in the range of 5.1%-10.6% (Table 1), higher than those of CPO. PPFO's inferior quality could be contributed by longer storage and exposure of PPMF to atmospheric conditions prior to extraction (Neoh *et al.*, 2011; Nur

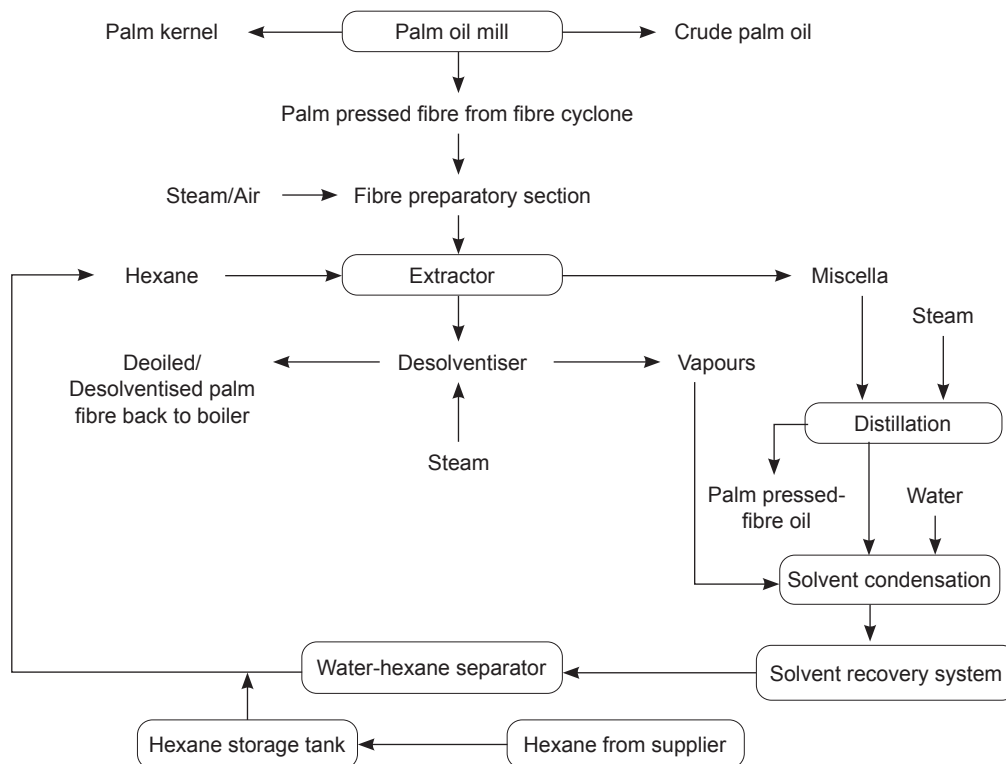


Figure 1. Process flow of solvent extraction plant.

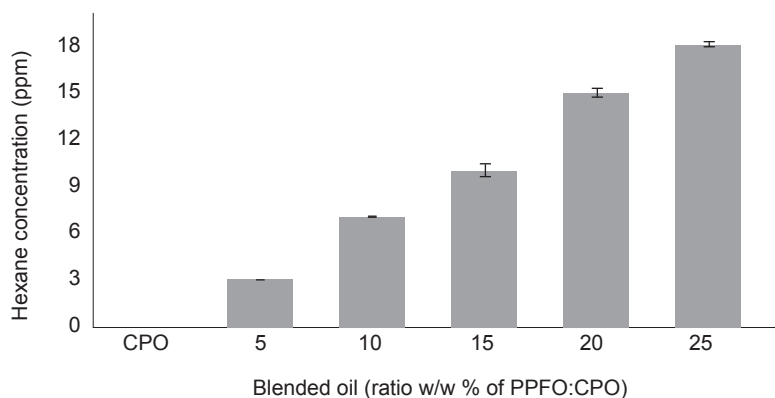


Figure 2. Hexane concentration of crude palm oil (CPO) blended with different dosage of palm pressed-fibre oil (PPFO). Results are mean values ($n=3$) by triplicate blending oil samples. Error bars represent the standard deviations of mean values.

Sulihatimarsyila *et al.*, 2019; Rusnani *et al.*, 2012). High extraction temperature also led to hydrolysis of the residual oil and thus, increased the FFA values (Bahadi *et al.*, 2016; Che Man, *et al.*, 1999; Japir *et al.*, 2017; Rusnani *et al.*, 2012). Table 1 exhibited that PPFO and CPO have various initial FFA values and theroretically correlated with the effects of physical blending on the quality properties. As a result, blending of PPFO with CPO gradually increased FFA values from $3.42 \pm 0.04\%$ to $3.91 \pm 0.06\%$ with the increased in PPFO from 0%-25% (w/w of CPO) as shown in Figure 3. This result indicates that FFA value would increase by 2% for every 5% (w/w) PPFO dosage into CPO.

Effect on Deterioration of Bleachability Index (DOBI)

The DOBI indicates the oxidation rate of the product and the ease of refining for the oil. High DOBI value implies that the fruit is fresh, ripe and free of contaminants. Results showed that DOBI values for PPFO were relatively lower than CPO, from 1.62 to 2.02 and 2.43 to 2.83, respectively (Table 1). The result was in agreement with other studies conducted by Umami Kalsum and Rohaya (2019) and Nur Sulihatimarsyila *et al.* (2019). DOBI value is one of the main indicators to show the deterioration of the oil. Good quality oil

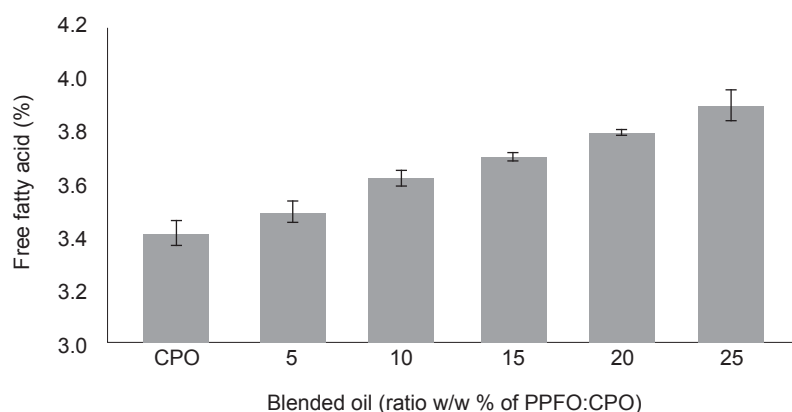


Figure 3. Free fatty acid (FFA) value of crude palm oil (CPO) blended with different dosage of palm pressed-fibre oil (PPFO). Results are mean values ($n=3$) by triplicate blending oil samples. Error bars represent the standard deviations of mean values.

has a DOBI value of more than 2.3 as specified in MS 814:2007 (Farah Khuwailah *et al.*, 2019). Overheating and oxidation of palm pressed fibre during processing could have contributed to low DOBI value (Rusnani *et al.*, 2012; Tan *et al.*, 2017). Other factors contributing to low DOBI values are prolonged storage before processing and contamination by other materials contained in the pressed fibre. Umami Kalsum and Rohaya (2019) suggested that the variation in DOBI values among the mills could be attributed to differences in operating parameters and mill practices for solvent extraction. Blending results showed that the DOBI values decreased from 2.76 ± 0.04 to 2.33 ± 0.03 with the increment of PPFO dosage (from 0%-25% w/w) into CPO as shown in Figure 4. These results showed that the decrease in DOBI values is proportional to the addition of PPFO into CPO. This blending result indicates that blending more than 25% (w/w) PPFO into CPO will lower DOBI values to less than 2.3, lower than the specification of PORAM in MS 814:2007 (Farah Khuwailah *et al.*, 2019).

Effect on Phosphorus Content

The phosphatides content in good quality CPO normally ranged between 10-20 ppm. Elemental analysis of phosphorus (P) indicated the presence of phospholipids in oil. Previous study showed that good quality CPO containing lower phosphorus content (below than 20 ppm) would exhibit very low level of trace metals, such as iron and copper. Ai (1990) reported that the ideal amount of phosphorus content for good quality CPO is 15 ppm. High acidity CPO which showed high phosphorus content, exceeding 30 ppm, would affect the oil stability which imparts undesirable level of impurities and gums trace metals such as iron and copper. Studies also showed that high amount of phospholipids would destroy the vitamin A in CPO (Goh *et al.*, 1984; Lau *et al.*, 2006; Rakmi *et al.*, 1983). It is thus, desirable to have low phospholipids levels in the blended oils for better oxidative stability and easier bleaching process. However, results from this study showed that PPFO contain significant

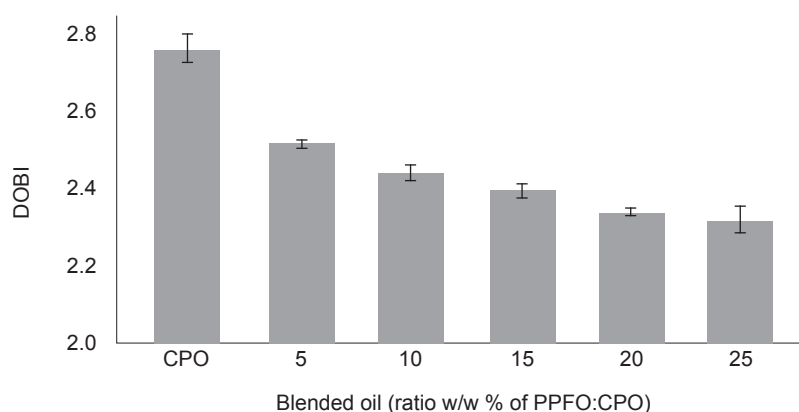


Figure 4. Deterioration of bleaching index (DOBI) of crude palm oil (CPO) blended with different dosage of palm pressed-fibre oil (PPFO). Results are mean values ($n=3$) by triplicate blending oil samples. Error bars represent the standard deviations of mean values.

amount of phosphorus (214-247 ppm), while CPO has lower phosphorus content (16-27 ppm) (Table 1). Umami Kalsum and Rohaya (2019) also reported phosphorus content of up to 1063 ppm in PPFO sample in one of the palm oil mills surveyed by MPOB (Table 1). As reported by Goh *et al.* (1982), oil from solvent extracted ripe fruits mesocarp fibre contains high amount of phospholipids (1000-2000 ppm) compared to commercial CPO (20-80 ppm). Results from this study showed that phosphorus content in blended oil increased gradually from 34.67 ± 3.48 ppm to 63.84 ± 4.02 ppm as the ratio of PPFO increased from 0%-25% (w/w of CPO) (Table 2). This shows that high PPFO dosage will increase the phosphorus level in CPO, which in turn, will complicate the CPO refining process. Consequently, high amount of bleaching earth is required, which subsequently incur high cost for refining processes (Lau *et al.*, 2013; Umami Kalsum and Rohaya, 2019). In addition, the initial phosphorus content in CPO and PPFO is crucial to ensure blending CPO with PPFO would not affect the downstream processing. This blending result suggested that phosphorus content will increase by 15% for every 5% (w/w) of PPFO blended into CPO.

Effect on Chlorine Content

Chloride ion was identified as one of the main precursor of 3-MCPD esters formation in refined palm oil (Freudenstein *et al.*, 2013; Ramli *et al.*, 2015). 3-MCPD esters is generally formed when acylglycerols in oil reacted with high temperature (260°C) in the presence of chloride ion during deodourisation in refining process (Parveez *et al.*, 2019; Ramli *et al.*, 2015). In a random survey led by MPOB, the total chlorine content in CPO from eight palm oil mills in 2018 and 2019 ranged from 1.95-10.89 ppm (Ramli *et al.*, 2015; Umami Kalsum and Rohaya, 2019). Several years later, Umami Kalsum and Rohaya (2019) reported that PPFO contains

23.41-127.23 ppm of chlorine as depicted in Table 1. Theoretically, physical blending of CPO with PPFO would result in increment of chlorine contents in the blended oil. The concentration of chlorine in the blended oils increased, with the increased in PPFO dosage (from 0%-25% w/w of CPO) with the results being in the range from 3.85-7.49 ppm. Consequently, this resulted in the increment of 3-MCPD esters formation in blended oil and in refined palm oil when heated at higher temperature (Ramli *et al.*, 2015). Study conducted by Umami and Rohaya (2019) is an evident that blending CPO with PPFO would increase 3-MCPD esters from 0.2-15 ppm when PPFO was added up to 10% (w/w) into CPO. In fact, in October 2018, the European commission suggested two maximum levels of 3-MCPD esters in refined vegetable oil, which are 1.25 ppm for oils and fats (coconut, maize, rapeseed, sunflower, soybean and palm kernel) and 2.5 ppm for other vegetables oil, fish oil and also mixture of oils and fats (Parveez *et al.*, 2019; Umami Kalsum and Rohaya, 2019).

Effect on Heavy Metal Content

Rakmi *et al.* (1983) reported that heavy metals such as iron and copper are active prooxidants that would accelerate the oxidation process in CPO. Heavy metals would also catalyse the decomposition of hydroperoxides to free radicals and hastened the oxidation process. Flider and Orthoefer (1981) reported that similar reaction was observed in other oils such as soybean oil where its quality deteriorated when iron and copper's concentrations were greater than 30 ppb and 5 ppb, respectively. Ames *et al.* (1960) also reported that high amount of iron content in CPO would complicate the bleaching process in refinery, while copper would increase the colour of refined palm oil from 2.0 to 4.8 Red Lovibond. Previous studies showed that maximum level of iron and copper in

TABLE 2. TRACE ELEMENTS CONTENT IN CRUDE PALM OIL (CPO), PALM PRESSED-FIBRE OIL (PPFO) AND BLENDED OILS (BO)

Samples	Trace elements concentration (ppm)			
	Iron (Fe)	Copper (Cu)	Phosphorus (P)	Chloride (Cl)
PPFO	45.09 ± 0.03	0.66 ± 0.02	228.05 ± 0.04	23.41 ± 0.03
CPO	13.58 ± 0.02	0.19 ± 0.03	34.67 ± 0.01	3.85 ± 0.02
BO5%PPFO	14.63 ± 0.02	0.22 ± 0.01	44.23 ± 0.03	5.13 ± 0.04
BO10%PPFO	15.45 ± 0.03	0.27 ± 0.02	45.72 ± 0.02	5.82 ± 0.03
BO15%PPFO	17.26 ± 0.04	0.30 ± 0.03	52.91 ± 0.02	6.49 ± 0.02
BO20%PPFO	15.96 ± 0.03	0.35 ± 0.02	57.31 ± 0.03	6.66 ± 0.03
BO25%PPFO	17.63 ± 0.02	0.38 ± 0.01	63.84 ± 0.02	7.49 ± 0.02

Note: Results are mean values (n=3) by triplicate blending oil samples.

refined palm oil to be exported to Australia were 500 ppb and 10 ppb, respectively (Berger, 1982; Rakmi *et al.*, 1983). The concentrations of heavy metals in PPFO, namely iron (45.09 ppm) and copper (0.66 ppm) were higher compared to CPO (Table 2). Blending CPO with PPFO would result in the increment of heavy metals concentrations in the blended oil. The concentration of iron and copper in the blended oils had increased, with the increase in PPFO dosage (from 0%-25% w/w of CPO) in the range from 13.58-17.63 ppm and 0.19-0.38 ppm, respectively. This result showed that high initial amount of heavy metals content in PPFO would effect the blended oils and increase by 5% for every 5% (w/w) PPFO dosage blended into CPO.

Effect on Iodine Value (IV)

IV is another important parameter that indicates the oil oxidative stability and for determining oil identities (Ayyildiz *et al.*, 2015; Talpur *et al.*, 2010). Determination of IV involved measuring the degree of unsaturation or double bonds in fats and oils. Higher IV indicates greater degree of unsaturation, hence, lower oxidative stability (Soek *et al.*, 2019). Table 1 shows that the IV of CPO was higher than PPFO with the values ranges from 51.96-53.22 and 49.11-51.22, respectively. The decrease in IV in PPFO can be attributed to the destruction of double bonds by oxidation or polymerisation. Comparing with CPO, the IV in blended oils decreased from 51.96 ± 0.37 to 50.21 ± 0.03 with the increased in PPFO dosage from 0% to 25% w/w of CPO (Figure 5). This indicated that there is an increase in saturated fatty acids (SFA) when CPO is blended with PPFO as this oil contained high SFA compositions (Table 4). This result is in agreement with findings by Soek *et al.* (2019) which indicated that IV in PPFO is lower than CPO, implying a higher saturation level in PPFO as compared to CPO.

Effect on Oil Oxidative Stability

Oxidative stability is a significant parameter in evaluating the shelf life of oils and fats. FAC and minor components such as tocopherols, FFA and IV could affect the oil oxidative stability (Ayyildiz *et al.*, 2015; Bozan and Temelli, 2008; Cao *et al.*, 2014). Oxidative stability is interrelated with the compositions of fatty acids, in which when more unsaturated fatty acids (UFA) are present in the edible oil, the oxidation reaction will occur faster (Liu and White, 1992; Magdalena *et al.*, 2018; Ramli *et al.*, 2017). CPO contains slightly higher UFA (51.96%) compared to PPFO (49.11%) (Table 4). UFA in palm oil comprises of C18:3 (linolenic acid), C18:2 (linoleic acid) and C18:1 (oleic acid). Previous studies have found that linolenic acid was the first to be oxidised, followed by linoleic acid and oleic acid (Liu and White, 1992; Magdalena *et al.*, 2018). CPO showed lower oxidative stability with shorter induction time (14-17 hr) in Rancimat test compared to PPFO (18-25 hr) (Table 1). As a result, blending PPFO with CPO stabilised the CPO, whereby the oxidative values of the blended oils varied from 14.31 ± 1.50 hr to 16.76 ± 0.74 hr. The oxidative stability of the blended oils of up to 25% (w/w) PPFO was comparable to CPO as depicted in Figure 6. PPFO contains of high amount of carotenes that consist of antioxidants properties. This would contribute to the increased stability of the blended oils.

Effect on Phytonutrients Content

Table 3 shows the phytonutrients content in CPO, PPFO and blended oils. PPFO that was commercially produced by solvent extraction at the palm oil mill contained higher amount of carotenes (1258-1750 ppm) compared to CPO (537-572 ppm) (Table 1). These results are in agreement

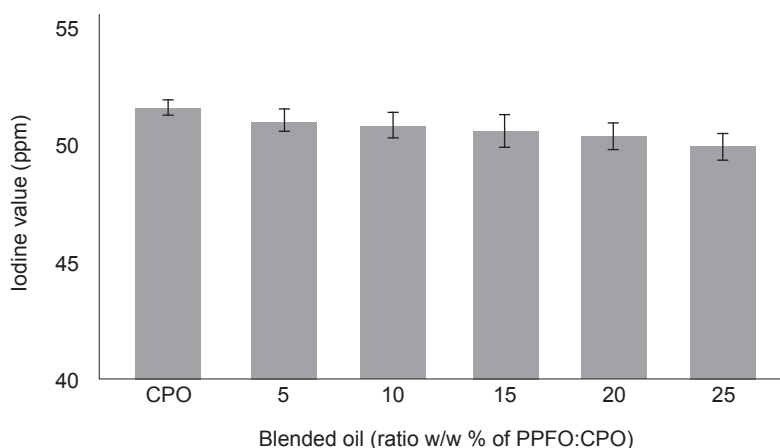


Figure 5. Iodine value (IV) of crude palm oil (CPO) blended with different dosage of palm pressed-fibre oil (PPFO). Results are mean values ($n=3$) by triplicate blending oil samples. Error bars represent the standard deviations of mean values.

TABLE 3. PHYTONUTRIENTS CONTENT IN CRUDE PALM OIL (CPO) PALM PRESSED-FIBRE OIL (PPFO) AND BLENDED OILS (BO)

Phytonutrients	CPO	PPFO	Blended oil (% w/w PPFO: CPO)				
			BO5%PPFO	BO10%PPFO	BO15%PPFO	BO20%PPFO	BO25%PPFO
Carotene (ppm)	539 ± 14	1 481 ± 27	554 ± 5	608 ± 68	609 ± 22	623 ± 14	654 ± 8
Sterol (ppm)	433 ± 8	1 529 ± 1	444 ± 25	497 ± 15	540 ± 33	629 ± 21	808 ± 17
Squalene (ppm)	475 ± 7	1 721 ± 10	780 ± 8	881 ± 5	962 ± 4	1 048 ± 7	1 201 ± 9
Vitamin E (ppm)	1 185 ± 64	1 609 ± 71	1 489 ± 9	1 494 ± 10	1 591 ± 22	1 607 ± 89	1 626 ± 92
Composition of tocopherol and tocotrienol (%)							
• α -tocopherol	5	24	5	5	6	6	7
• α , β , γ , δ -tocotrienols	95	76	95	95	94	94	93

Note: Results are mean values (n=3) by triplicate blending oil samples.

TABLE 4. COMPARISON OF FATTY ACID COMPOSITIONS AND ACYLGlycerIDES OF CRUDE PALM OIL (CPO), PALM PRESSED-FIBRE OIL (PPFO) AND BLENDED OILS

	CPO	PPFO	Blended oil (% w/w PPFO: CPO)					MS 814:2007
			BO5%PPFO	BO10%PPFO	BO15%PPFO	BO20%PPFO	BO25%PPFO	
Fatty acid compositions (%)								
Saturated fatty acids (SFA)								
C12:0	0.19 ± 0.02	1.33 ± 0.05	0.24 ± 0.01	0.28 ± 0.01	0.34 ± 0.01	0.39 ± 0.02	0.42 ± 0.03	0.1 – 0.4
C14:0	1.00 ± 0.02	1.34 ± 0.01	1.00 ± 0.02	1.02 ± 0.00	1.03 ± 0.02	1.05 ± 0.01	1.07 ± 0.01	1.0 – 1.4
C16:0	45.69 ± 0.09	41.22 ± 0.02	45.56 ± 0.21	45.16 ± 0.14	44.82 ± 0.04	44.63 ± 0.05	44.44 ± 0.02	40.9 – 47.5
C18:0	3.97 ± 0.08	4.09 ± 0.06	4.18 ± 0.17	4.10 ± 0.03	4.07 ± 0.03	4.09 ± 0.03	4.13 ± 0.04	3.8 – 4.8
Unsaturated fatty acids (UFA)								
C18:1	41.96 ± 0.01	39.24 ± 0.08	39.30 ± 0.27	39.55 ± 0.08	39.57 ± 0.09	39.69 ± 0.08	39.77 ± 0.01	36.4 – 41.2
C18:2	10.00 ± 0.08	9.87 ± 0.02	9.70 ± 0.10	9.87 ± 0.07	9.92 ± 0.04	9.89 ± 0.03	9.90 ± 0.02	9.2 – 11.6
Acylglycerides (%)								
MAG/FFA	5.06 ± 0.01	11.44 ± 0.01	5.38 ± 0.01	5.61 ± 0.03	5.84 ± 0.02	6.05 ± 0.05	6.28 ± 0.00	N/A
DAG	5.58 ± 0.03	9.51 ± 0.06	5.74 ± 0.08	5.88 ± 0.02	6.03 ± 0.02	6.30 ± 0.02	6.32 ± 0.03	N/A
TAG	89.36 ± 0.07	79.05 ± 0.04	88.89 ± 0.03	88.52 ± 0.02	88.13 ± 0.02	87.74 ± 0.06	87.44 ± 0.02	N/A

Note: MAG - monoacylglycerols; DAG - diacylglycerols; TAG - triacylglycerols; FFA - free fatty acid; BO - blended oil; N/A - not available. Results are mean values (n=3) by triplicate blending oil samples.

with findings by Umami Kalsum and Rohaya (2019), who reported carotenes content in PPFO can be up to 1913 ppm. The carotenes content in blended oils increased from 539 ± 14 ppm to 654 ± 8 ppm with the increment in PPFO dosage (from 0%-25% w/w of CPO). Vitamin E (tocopherols and tocotrienols) in palm oil comprises of four major isomers: α -tocopherol, α -tocotrienol, γ -tocotrienol and δ -tocotrienol. Vitamin E is well-known for its antioxidant and nutritional values (Lau *et al.*, 2007; 2008; Neoh *et al.*, 2011; Ng and Choo, 2011; Nur Sulihatimarsyila *et al.*, 2019; Rusnani *et al.*, 2012). Table 3 shows that PPFO has higher amount of vitamin E (1609 ± 71 ppm) compared to CPO (1185 ± 64 ppm). The α -tocopherol content in PPFO was higher (24% of total tocopherols) than CPO (5% of total tocopherols) (Table 3). These results are consistent with

data obtained from previous findings (Choo *et al.*, 1996; Goh *et al.*, 1985; Ng and Choo, 2011; Rusnani *et al.*, 2012). Results also showed that the composition of tocotrienols in PPFO, which comprised of α -tocotrienol, γ -tocotrienol and δ -tocotrienol, was lower (76% of total tocopherols than CPO (95% of total tocopherols). However, blending CPO with PPFO affected the tocotrienols and tocopherol compositions in blended oils (Table 5). Compositions of tocopherol in blended oils increased from 5%-7% of total tocopherols while tocotrienols composition decreased from 95%-93% of total tocopherols with the increment in PPFO dosage from 0%-25% (w/w), respectively. PPFO is also rich in sterols and squalene (Table 3). The concentration of sterols in PPFO (1529 ± 1 ppm) was higher than CPO (433 ± 8 ppm). Squalene content in PPFO (1721 ± 10 ppm) was higher than CPO

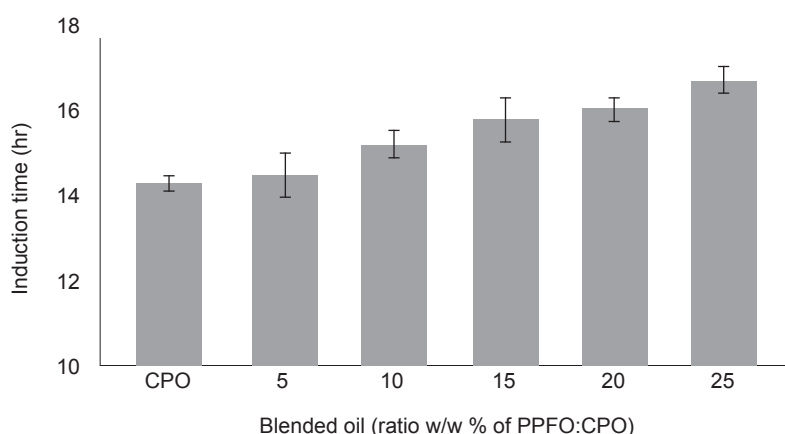


Figure 6. Oxidative stability of crude palm oil (CPO) blended with different dosage of palm pressed-fibre oil (PPFO). Results are mean values ($n=3$) by triplicate blending oil samples. Error bars represent the standard deviations of mean values.

(475 ± 7 ppm). Blending of CPO with PPFO from 0%-25% (w/w) dosage increased the concentrations of sterols and squalene in the blended oils, from 433 ± 8 ppm to 808 ± 17 ppm and 475 ± 7 ppm to 1201 ± 9 ppm, respectively.

Effect on Fatty Acid Compositions (FAC) and Acylglycerides

PPMF from the palm oil mill consists of a mixture of palm mesocarp fibre, shell, crushed kernel and debris. Hence, the oil recovered from this mixture has the characteristics of all these products (Lau *et al.*, 2006; Rusnani *et al.*, 2012). The nutritional status of palm oil is determined by the types of fatty acids contained in palm oil, which consists approximately 50% SFA with 45% palmitic acid (C16:0), 5% stearic acid (C18:0), and trace amount of myristic acid (C14:0) and lauric acid (C12:0) (Sambanthamurthi *et al.*, 2000). The UFA consist of approximately 40% oleic acid (C18:1), and 10% polyunsaturated linoleic acid (C18:2) and linolenic acid (C18:3) (Noh *et al.*, 2002; Sambanthamurthi *et al.*, 2000). Table 4 shows the comparison of FAC in PPFO and CPO. The FAC of PPFO is quite similar to CPO with palmitic ($41.22 \pm 0.02\%$) and oleic acids ($39.24 \pm 0.08\%$) being the major fatty acids. However, composition of lauric acid in PPFO was significantly higher than CPO ($1.33 \pm 0.05\%$). Presence of lauric acid in PPFO had attributed towards contamination of kernel oil as the pressed fibre comprised of mixture of broken nuts and kernels, discharged together with mesocarp fibre after mechanical pressing (Neoh *et al.*, 2011; Rusnani *et al.*, 2012). According to Siew and Berger (1981), lauric acid is present in palm kernel oil with a composition of 48%, which discriminates palm kernel oil from CPO. FAC of the blended oils samples were comparable to those of CPO and remained in the range specified by PORAM in MS 814:2007 (Table 4). However, composition of palmitic acid decreased slightly

from $45.69 \pm 0.09\%$ to $44.44 \pm 0.02\%$ with the 0%-25% (w/w) PPFO dosage in CPO. Nevertheless, the compositions of lauric, myristic and oleic acids in blended oils increased gradually with the increase in PPFO dosages in CPO, at $0.19 \pm 0.02\%$ to $0.42 \pm 0.03\%$, $1.00 \pm 0.02\%$ to $1.07 \pm 0.01\%$ and $39.24 \pm 0.08\%$ to $39.77 \pm 0.01\%$, respectively. CPO constitutes mainly 90%-98% TAG, 2%-6% DAG, 2%-5% MAG/FFA and minor composition of phytonutrients (Neoh *et al.*, 2011). Results indicated that PPFO has higher DAG, $9.51 \pm 0.06\%$, compared to CPO ($5.58 \pm 0.03\%$). On the other hand, the TAG content in PPFO ($79.05 \pm 0.04\%$) was lower than CPO ($89.36 \pm 0.07\%$) (Table 4). It was found that the compositions of DAG in blended oils with PPFO dosage from 0%-25% increased slightly, ranging from $5.58 \pm 0.03\%$ to $6.32 \pm 0.03\%$, whilst TAG decreased from $89.36 \pm 0.07\%$ to $87.44 \pm 0.02\%$.

CONCLUSION

This study has shown that blending CPO with PPFO would deteriorate the original CPO quality, by lowering DOBI and IV and increase levels of phosphorus, FFA, hexane and trace metals, even at low dosage of PPFO. Results indicated that PPFO produced from the commercial solvent extraction plant shows various initial properties. Blending results depicted that physical blending properties would be dependent on initial properties of PPFO and CPO. Furthermore, this blending analysis shows an increment in chloride content, which is one of the main precursors of 3-MCPD esters formation. Thus, it is not recommended to blend PPFO into CPO, as the blended oil deteriorate CPO quality. Nevertheless, PPFO has better quality than CPO in terms of phytonutrient contents, signifying its potential in other applications. Instead of blending PPFO with CPO, it is suggested for PPFO to be classified as valuable oil and sold as pharma-

industrial grade oil since it is rich in phytonutrient and it is suitable for other applications such as pharmaceutical industry and biodiesel.

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