# PROPERTIES OF RESIDUAL PALM PRESSED FIBRE OIL

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# ABSTRACT

Residual fibre oil was recovered from palm pressed fibre by using a solvent (hexane). The oil was analysed for its physical and chemical properties. The macro-nutrient and element contents of the pressed fibre were also analysed. The residual fibre oil contained high amounts of phosphorus (144 ppm), vitamin E (1153 ppm) and carotenes (1877 ppm). The compositional content of vitamin E present was 54.0%  $\alpha$ -tocopherol, 19.2%  $\alpha$ -tocotrienol, 16.7%  $\gamma$ -tocotrienol and 10.1%  $\delta$ -tocotrienol. The major fatty acids of the residual fibre oil were palmitic and oleic acid at 31.9% and 24.8%, respectively. The oil also contained a high amount of lauric acid (22.0%) which is the major fatty acid in palm kernel oil. Density of the residual fibre oil as a function of temperature was measured at temperatures ranging from 40°C to 75°C. Density exhibited a linear relationship with temperature. Viscosity of the residual fibre oil was shown to decrease with an increase in temperature. The formation of needle-shaped crystals was observed when the residual oil was cooled down from 36°C to 34°C. There were no significant changes in the macro-nutrient and element contents of the palm pressed fibre before and after oil extraction with hexane.

Keywords: palm pressed fibre, residual fibre oil, properties, density, viscosity.

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#### INTRODUCTION

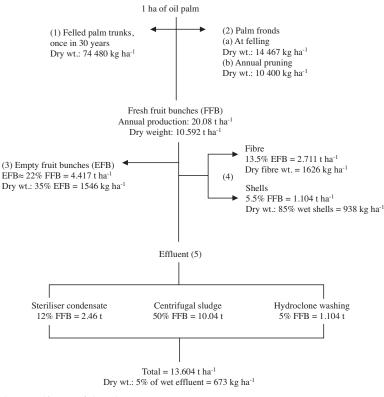
Fresh fruit bunches (FFB) are processed in the palm oil mill to produce two types of products: crude palm oil (CPO) and palm kernels. Palm oil milling processes consist of various stages which include sterilisation of the FFB, stripping, pressing, clarification and purification of the crude oil, nut cracking and kernel recovery. Besides the two main products, the mill also generates about 1.5 t of empty fruit bunches, 1.6 t of palm pressed fibre, 0.9 t of palm shells, 2.4 t of steriliser condensate, 10.0 t of centrifugal sludge and 0.7 t of dry mill effluent per hectare of oil palm annually as illustrated in *Figure 1*. Besides being used as a solid fuel for boiler

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Oil losses occur at various stages of processing in a palm oil mill. In the pressing stage, oil is 'lost' in the pressed fibre. The amount of oil entrapped in these fibre depends very much on the effect of sterilisation on the fruit, the conditioning of the sterilised fruits in the digester, and also the pressure exerted on the press cake during pressing. Oil losses in the pressed fibre range from 5%-8% on a dry weight basis. The utilisation of palm pressed fibre as well as the residual fibre oil recovered from the fibre will be able to change the by-products into valuable products.

As palm oil is well-known for its versatility in both edible and non-edible applications, great efforts are currently undertaken to study the application of the by-products generated from the palm oil milling process. These by-product applications require extensive studies on the physico-chemical properties of the by-products in order to ensure their suitability as raw materials for various final



Source: Chan *et al*. (1981).

Figure 1. Diagrammatic representation of actual quantities of oil palm wastes.

products. Some research related to the extraction of minor components from palm pressed fibre has been carried out by Choo *et al.* (1996). The present work covers the analysis of the physical properties of residual fibre oil, including the chemical contents of some of the minor components, *viz.* carotene, vitamin E and phosphorus. This article also presents the macro-nutrient and element contents in palm pressed fibre before and after hexane-extraction.

### MATERIALS AND METHODS

Palm pressed fibre and CPO samples were collected from a palm oil mill. About 10 kg of palm pressed fibre and 500 ml of CPO were collected over three consecutive weeks. The palm pressed fibre sample was first dried in an oven at 60°C, and the residual fibre oil was later hexane-extracted from the sample using soxhlet apparatus. The solvent from the oil/hexane mixture was removed using a rotary evaporator to obtain the residual fibre oil. All solvents and chemicals which were of chromatographic and analytical grades were purchased from Merck (Germany), J T Baker (Germany) and Sigma (Germany).

The oil samples were analysed according to the MPOB Test Methods (2005) for phosphorus, carotene, density, free fatty acids, peroxide value and fatty acid composition. Viscosity was determined by using a capillary U-tube viscometer, and the parameter was measured at different temperatures. Vitamin E content was determined by high performance liquid chromatography (HPLC) and a fluorescence detector (Agilent Technologies) with a C18 column (150 mm × 4.6 mm i.d,), and at a flowrate of 1.0 ml min<sup>-1</sup> of acetonitrile/methanol (50:50, v/v). The crystal structure of residual fibre oil when melted at declining temperatures from 40°C was observed through an optical microscope, Model Leica DMLP (Leica, Germany).

The macro-nutrient content of palm pressed fibre was determined using an atomic absorption spectrometer (Perkin Elmer, Model Analysis 200). The morphology and the elemental content of the fibre were determined using a scanning electron micrograph (Hitachi S3400 SEM/EDX).

#### **RESULTS AND DISCUSSION**

#### Vitamin E

The compositional content of vitamin E in residual fibre oil is shown in *Table 1*. The residual oil contains a higher amount of  $\alpha$ -tocopherol than CPO. The vitamin E content comprises 623 ppm (54.0%)  $\alpha$ -tocopherol, 221 ppm (19.2%)  $\alpha$ -tocotrienol, 192 ppm (16.7%)  $\gamma$ -tocotrienol and 117 ppm (10.1%)  $\delta$ -tocotrienol. CPO is rich in  $\gamma$ -tocotrienol, having a vitamin E composition as follows: 141 ppm (21.1%)  $\alpha$ -tocopherol, 169 ppm (25.3%)  $\alpha$ -tocotrienol, 247

Composition	Residual fibre oil			Crude palm oil		
	Range	Mean	SD	Range	Mean	SD
α-tocopherol	519-765	623	97	133-147	141	6
α-tocotrienol	194-245	221	21	154-200	169	9
γ-tocotrienol	171-214	192	16	234-268	247	11
δ-tocotrienol	79-140	117	21	106-125	112	8
Total	900-1 200			600-800		

TABLE 1. TOCOPHEROL AND TOCOTRIENOLS IN RESIDUAL FIBRE OIL AND CRUDE PALM OIL (ppm)

Note: SD = standard deviation.

TABLE 2. PHOSPHORUS AND CAROTENES IN RESIDUAL FIBRE OIL AND CRUDE PALM OIL

	Residual fibre oil			Crude palm oil		
	Range	Mean	SD	Range	Mean	SD
Phosporus (ppm)	138-149	144	5	13-16	15	1.5
Carotenes (ppm)	1 716-2 083	1 877	134	599-619	608	8

Note: SD = standard deviation.

ppm (36.9%)  $\gamma$ -tocotrienol and 112 ppm (16.7%)  $\delta$ -tocotrienol. The total concentration of tocopherol and tocotrienols in the residual fibre oil was 1153 ppm which makes the oil a valuable source of vitamin E. Vitamin E, a generic name for the family of tocopherols and tocotrienols, is well-known for its antioxidant and nutritional values (Burton and Ingold, 1989; Qureshi *et al.*, 1991; Kamal-Eldin and Appelqvist, 1996; Yu *et al.*, 1999; Nafeeza *et al.*, 2000; Nesaretnam *et al.*, 2000).

#### Phosphorus

Residual fibre oil and CPO contained about 144 and 15 ppm of phosphorus, respectively (Table 2). Goh et al. (1982) found that phosphorus content in CPO is low, but a substantial amount of phosphorus is present in palm pressed fibre and sludge. High phospholipid or phosphorus content has been suspected to have detrimental effects on oil quality (Chooi and Koh, 1981; Jacobsberg, 1983). Generally, phospholipids in CPO are eliminated during the refining process through the degumming and bleaching steps. However, phospholipids can also be a valuable minor component because they have been reported to show antioxidant effects (Jacobsberg, 1983; Keyong et al., 2009). Phospholipids, commercially known as lecithin, have potential as a multifunctional additive for food, pharmaceutical and industrial applications (Endre and Szuhaj, 1996). Vegetable lecithin is commercially produced from soyabean and rapeseed (van Nieuwenhuyzen and Tomás, 2008).

#### Carotenes

*Table 2* shows that residual fibre oil contained a higher amount of carotenes (1877 ppm) than

CPO (608 ppm). Visually, the deep red colour of residual fibre oil confirmed the presence of the higher amount of carotenes. In CPO, carotenes are normally removed during the refining process by bleaching earths and by the thermal destruction of the carotenes at high temperatures with the aim of producing a light yellow refined palm oil. Carotenes are well-known for having provitamin A activity, with  $\beta$ -carotene recording the highest activity. CPO is reported to have 15 to 300 times more retinol (provitamin A) equivalent than carrot, leafy green vegetables and tomato (Choo, 1994). Therefore, the large amount of carotenes in residual fibre oil provides another source of carotene for producing a nutraceutical product.

## Density

Figure 2 illustrates the density of residual fibre oil as a function of temperature. At 50°C, the density of residual fibre oil is 0.8692 kg litre<sup>-1</sup>, which is slightly lower than the density of CPO  $(0.8892 \text{ kg litre}^{-1})$  at the same temperature (Tan *et* al., 2000). The density of the residual fibre oil was found to decrease linearly with temperature within the studied temperature range of 40°C to 75°C. A study by Rice (1989) concluded that the variation in density of palm oil with temperature is linear over the temperature range from 39°C to 200°C. A similar linear relationship between density and temperature has also been observed by Coupland and McClements (1997). The linear regression equation obtained for the residual fibre oil is as follows:

$$y = -0.0007x + 0.9053 (R^2 = 0.9979)$$
 Eqn. 1

where *y* is density in kg litre<sup>-1</sup> and *x* is temperature in °C.

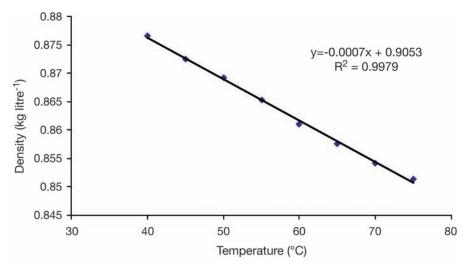


Figure 2. Density of residual fibre oil in relation to temperature.

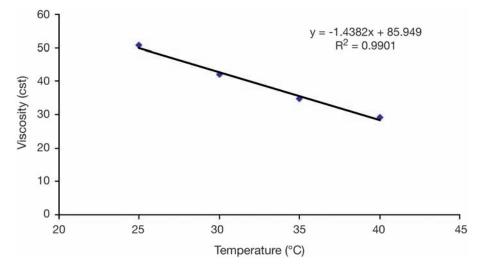


Figure 3. Viscosity of residual fibre oil in relation to temperature.

#### Viscosity

Viscosity is defined as the resistance of a fluid to flow. It is one of the important parameters in the design of process equipment for many industries including oils and fats. The viscosity of residual fibre oil as a function of temperature is shown in *Figure 3*. Over the temperature ranging from 25°C to 40°C, the viscosity was found to decrease linearly with increasing temperature, and the following regression equation was obtained:

$$y = -1.4382x + 85.949 (R^2 = 0.9901)$$
 Eqn. 2

where *y* is viscosity in cst (centistokes) and *x* is temperature in  $^{\circ}$ C.

The negative association of viscosity with temperature has also been observed on other oils (Kapseu *et al.*, 1991; Noureddini *et al.*, 1992; Topallar and Bayrak, 1998; Eromosele and Paschal, 2003). The temperature-dependent characteristic of viscosity due to cohesive (intermolecular) forces between the liquid molecules has been explained by Munson *et al.* (1994). As temperature increases, these cohesive forces between the molecules decrease, and this leads to easier flow for the fluid.

# Fatty Acid Composition

*Table 3* shows the fatty acid composition of residual fibre oil and CPO. The major fatty acids in residual fibre oil were palmitic acid (31.9%), oleic acid (24.8%) and lauric acid (22.0%). The fatty acid composition of residual fibre oil was quite similar to that of CPO which had as major fatty acids palmitic acid (39.7%) and oleic acid (37.6%). A study by Siew and Berger (1981) on the chemical and physical

Fatty acid	<b>Residual fibre oil (%)</b>			CPO (%)		
	Range	Mean	SD	Range	Mean	SD
C10:0 caprylic	1.3-1.6	1.4	0.18	0	0	0
C12:0 lauric	20.0-23.6	22.0	1.82	0.2-0.3	0.3	0.05
C14:0 myristic	7.9-9.5	8.7	0.84	1.1-1.2	1.1	0.05
C16:0 palmitic	30.9-32.6	31.9	0.91	39.5-39.8	39.7	0.17
C16:1 palmitoleic	0	0	0	0.2-0.2	0.2	0.02
C18:0 stearic	3.6-5.7	4.3	1.20	7.6-11.0	8.8	1.93
C18:1 oleic	24.5-25.1	24.8	0.31	35.6-38.7	37.6	1.78
C18:2 linoleic	6.2-6.4	6.3	0.09	11.4-11.4	11.5	0.09
C18:3 (1) linolineic	0.2-0.4	0.3	0.08	0.3-0.3	0.3	0.01
C18:3 (2)	0.1-0.4	0.2	0.17	0.4-0.4	0.4	0.01
C18:3 (3)	0.1-0.3	0.1	0.13	0.2-0.2	0.2	0
Saturated		68.3			49.9	
Unsaturated		31.7			50.2	

TABLE 3. FATTY ACID COMPOSITION OF RESIDUAL FIBRE OIL AND CRUDE PALM OIL (CPO)

Note: SD = standard deviation.

TABLE 4. MACRO-NUTRIENT CONTENT IN PALM PRESSED FIBRE

	N	P	K	Mg	Ca
	(%)	(%)	(%)	(%)	(%)
Before extraction	$0.81 \pm 0.01$	$0.013 \pm 0.009$	$0.51 \pm 0.07$	$0.13 \pm 0.01$	$0.24 \pm 0.04$
After extraction	$0.89 \pm 0.11$	$0.004 \pm 0.005$	$0.40 \pm 0.05$	$0.12 \pm 0.01$	$0.21 \pm 0.01$

characteristics of Malaysian palm kernel oil showed that lauric acid constitutes about 48% of the total fatty acids in palm kernel oil. The presence of this short-chain fatty acid differentiates palm kernel oil from CPO. During mill processing, mechanical pressing is not able to extract 100% of oil from the mesocarp. Some amount of oil is 'lost' from being trapped within the mesocarp fibre. At the same time, some nuts and kernels will be broken during pressing. During nut/fibre separation, some broken nuts and kernels may go into the fibre stream. The palm kernel oil from these will be extracted together with palm oil during solvent extraction, thus contributing to some amount of lauric acid in the residual fibre oil.

## Crystallisation

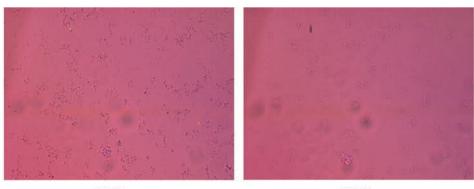
The crystal structure of residual fibre oil when the oil was cooled from 40°C to 30°C is shown in *Figure 4*. It was observed that no crystal was formed when the oil was cooled from 40°C to 36°C. However, needle-shaped crystals started to form when observed under the optical microscope as temperature dropped to 34°C. This oil crystallisation behaviour is very important in oil processing, especially during filtration and centrifugation (Timms, 1989). Filtering the residual fibre oil below 34°C will cause difficulty as the formation of more crystals will lead to a blockage problem and result in slower filtration. The filtration process can occur faster when the oil is heated so as to allow the oil components with a higher melting point to pass through the filter.

#### Free Fatty Acids and Peroxide Value

Fatty acids are the basic components of glycerides. Complete hydrolysis of triglycerides results in the formation of the free fatty acids and glycerols. Hydroperoxide is one of the first products formed by oxidation and is measured by the peroxide value. The free fatty acid and peroxide values in residual fibre oil were 31.4% and 8.5%, respectively. These high values were probably due to over-drying of the palm pressed fibre prior to extracting the residual fibre oil, and/or over-exposure of the samples to atmospheric conditions. Reactions leading to quality deterioration will occur faster at a higher temperature and over a longer storage period (Chong, 1991).

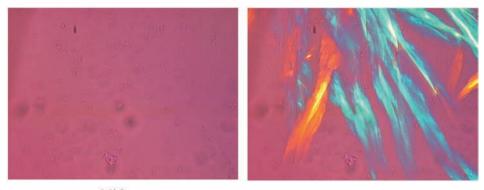
# Macro-nutrient and Element Contents Before and After Oil Extraction

The contents of macro-nutrients in palm pressed fibre before and after hexane-extraction are shown in *Table 4*. The macro-nutrient contents of palm pressed fibre before hexane-extraction are as follows: 0.81% N, 0.51% K, 0.24% Ca, 0.13% Mg and 0.013% P. It was found that there was no significant difference in the macro-nutrient contents of the fibre samples before and after extraction. These results indicate that extracting pressed fibre with hexane



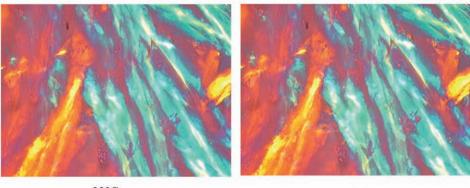


38°C



36°C

34°C



32°C

30°C Figure 4. Crystal structure of residual fibre oil at different temperatures.

TARIES	FIEMENT	CONTENT	IN PAIM	PRESSED FIBRE

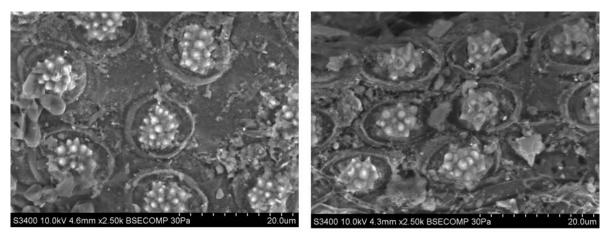
	C	O	A1	Si
	(%)	(%)	(%)	(%)
Before extraction	52.53	41.38	0.90	5.19
After extraction	50.55	42.53	1.31	5.40

had no significant effect on the macro-nutrient contents. Thus, the palm pressed fibre after hexaneextraction can still be used for other applications.

Table 5 shows the element contents in palm pressed fibre as determined using a SEM/EDX instrument. The results show that there was no significant change in element content when the fibre was subjected to hexane. The morphology of the pressed fibre showed that the structure of the fibre after hexane-extraction had more void areas than the fibre before solvent extraction (Figure 5). Hexane probably removed or loosened the oilbearing cells which were previously attached to the surface of or were within the fibre.

# **CONCLUSION**

Palm pressed fibre is easily available at palm oil mills, and the residual fibre oil can be recovered using hexane. The properties of residual fibre oil such as viscosity, density and crystallisation provide useful information for designing unit operations in the oils and fats industry. Residual fibre oil contains



Before extraction

After extraction

Figure 5. Morphology of palm pressed fibre.

a higher amount of valuable minor components such as carotenes, vitamin E (tocopherols and tocotrienols) and phospholipids as compared to CPO. The free fatty acid and peroxide values in residual fibre oil are high, making the residual oil suitable for non-edible applications. However, the valuable minor components such as phospholipids, vitamin E and carotenes in the residual fibre oil can be recovered or extracted for the production of nutraceutical products. With no significant difference in macro-nutrient and element contents in the palm pressed fibre before and after hexaneextraction, the fibre can also be used for other applications.

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