Recovered Oil from Palm-Pressed Fiber: A Good Source of Natural Carotenoids, Vitamin E, and Sterols

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ABSTRACT: Recovered fiber from pressed palm fruits, which is normally burned as fuel to provide energy for the palm oil mills, has now been found to be a rich source of carotenoids, vitamin E (tocopherol and tocotrienols), and sterols. Residual oil (5–6% on dry basis) extracted from palm press fibers contains a significant quantity of carotenoids (4000–6000 ppm), vitamin E (2400–3500 ppm), and sterols (4500–8500 ppm). The major identified carotenoids are α-carotene (19.5%), β-carotene (31.0%), lycopene (14.1%), and phytoene (11.9%). In terms of vitamin E, α-tocopherol constitutes about 61% of the total vitamin E present, the rest being tocotrienols (α-, γ-, and δ-). The major sterols present are β-sitosterol (47%), campesterol (24%), and stigmasterol (15%). The oil extracted from palm-pressed fiber is contaminated with about 30% of palm kernel oil. The quality of this fiber oil is slightly lower than that of crude palm oil in terms of the content of free fatty acids, peroxide value, and anisidine value.
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KEY WORDS: Carotenoids, fiber oil, lycopene, palm-pressed fiber, phytoene, phytofluene, sterols, tocopherol, tocotrienols.

An oil palm fruit bunch consists of two main parts, the stalk and the fruitlets. The fruitlets are made up of pericarp (i.e., mesocarp and exocarp) and the nut. Two types of oil can be obtained from the fruits, namely, palm oil from the oil cells in the mesocarp, and the kernel oil from the seed of the nut (kernel). These oils are extracted and recovered separately in palm oil mills and kernel crusher plants. In general, a palm oil mill extracts 20% of oil from the fresh fruit bunch and produces 23% empty bunch, 15% fiber, and 12% nut. Oil losses occur in various by-products, including the fiber, which remains after the mesocarp oil is extracted by a screw press. This fiber is referred to as palm-pressed fiber and contains 5–6% residual oil (on dry basis), but the fiber is normally burned as fuel to provide energy for the mill. Because extraction is conventionally done by single-stage pressing, the oil loss is considerable.

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In the present study, the extraction of oil and determination of valuable minor components from palm-pressed fiber are described. The fatty acid composition, as well as quality parameters of the palm-pressed fiber oil, are also discussed.

EXPERIMENTAL PROCEDURES

Extraction of residual oil from pressed fiber. Samples of palm-pressed fiber, freshly collected from oil palm mills, were dried at 50–60°C for 1 h, and the residual oil was extracted with different solvents, such as hexane, chloroform, and ethanol, in a Soxhlet apparatus. The residual oil recovered was weighed after rotary evaporation to remove solvents and pump-dried under vacuum. High-pressure Soxhlet extraction was also used to extract the residual oil from fibers with liquid CO_2 as an extraction medium at a pressure of 700–750 psi for 2 h.

High-performance liquid chromatography (HPLC) analysis of carotenes. About 5 g of the oil extracts was saponified with 5 mL of 50% aqueous KOH and heated at 50°C on a water bath in the dark under a stream of nitrogen for 45 min. The saponified sample was then extracted with 50-mL portions of petroleum ether until the extract became colorless. The combined petroleum ether extracts were washed four times with 50-mL portions of distilled water and dried over sodium sulfate. A portion of the extract was brought to dryness in a rotary evaporator at 30°C. The residue was dissolved in a known volume of the mobile phase, 100 μL of which was injected into the HPLC.

Analysis and detection of carotenes were carried out in a Varian 5000 HPLC instrument (Walnut Creek, CA), equipped with a variable-wavelength (190–990 nm) ultraviolet (UV)-100 detector and an SP 4270 integrator. Detection was recorded at different wavelength maxima of the carotenes and attenuated for the display of the various types of carotenes present.

The isocratic separation was performed on a ZORBAX ODS column (4.6 mm i.d. × 25 cm, stainless-steel, 5 μm spherical particles; DuPont Japan Ltd., Tokyo, Japan) protected with a DuPont guard column (20 μm ZORBAX ODS). A solvent system of acetonitrile (89%) and methylene chloride (11%) was used, and the flow rate was 1 mL/min.

Individual separated carotenes, eluting from the HPLC, were collected, and the absorbance spectra were recorded with a Hitachi (Tokyo, Japan) 150-20 spectrophotometer. The total carotenoid content was determined spectrophotometrically at 446 nm as described previously (1).

HPLC analysis of tocopherols and tocotrienols. Detailed analysis of the tocopherol and tocotrienols content in various oil samples was carried out by normal-phase HPLC with a 25 × 0.46 cm Licrosorp analytical column (Merck, Darmstadt, Germany), protected by a guard column (1.5 × 0.46 cm, 10 μm). A solvent system of 99:1 (hexane/2-propanol, vol/vol) with a flow rate of 0.8 mL/min was applied to the system, and the components were detected with a UV-visible detector set at a wavelength of 295 nm. All oil samples were pretreated according to the method described by Tan and Brzuskiewicz (2) by dissolving the oil in hexane and chilling overnight at −20°C. The samples were then centrifuged at 4°C to remove the precipitate, and the lipid-soluble fraction was chromatographed.

Determination of sterols. The sterol composition was determined by following the method of Peto *et al.* (3) with some modifications. Basically, the total sterols from the unsaponifiable matter were isolated by preparative thin-layer chromatography (TLC) with a solvent system of chloroform/diethyl ether/acetic acid 99:5:1 (vol/vol/vol). The sterol was extracted with diethyl ether, and the composition of various types of sterols was determined by gas chromatography with a 5-ft 3% OV-17 glass column. Cholesterol was used as an external standard for quantitative determinations.

RESULTS AND DISCUSSION

Samples of palm pressed fiber, collected from several palm oil mills, were subjected to various types of solvent extraction for about 1 h after drying at 50–60°C. The solvents used included hexane, chloroform, and liquid CO_2. The results show that the fiber oils extracted by these solvents contain higher concentrations of carotenoids, vitamin E, and sterols than normal commercial samples (Table 1).

Carotenoids. The carotenoid content of the oil extracted from fibers collected from various commercial palm oil mills ranges between 4000 and 6000 ppm, as shown in Table 2. Oil extracted from the pressed fiber of hybrid palm fruits contains a higher concentration (5000–7000 ppm) of carotenoids.

TABLE 1
Minor Components from Pressed Fiber and Crude Palm Oil

Sample of palm-pressed fiber oil	Carotenoid (ppm)	Tocopherol and tocotrienol (ppm)	Sterol (ppm)
Chloroform extracts	3800–5300	1650–2600	6906–8200
Hexane extracts	4000–5500	1200–2400	7050–8490
Liquid carbon dioxide extracts	4100–6000	2500–3000	4509–5200
Palm pressed fiber oil from hybrid palms	5000–7000	—	—
Crude palm oil[a]	500–700	600–1000	250–650

[a]Commercial Malaysian crude palm oil.

TABLE 2
Carotenoid Content of Residual Oils from Fibers Collected from Various Palm Oil Mills

Palm oil mills	Carotenoid (ppm)
Mill A[a]	4070
Mill B	4100–4520
Mill C	4000–5000
Mill D	4000–5000
Mill E	3600–4770
Mill F	4440–5050

[a]Except for a single sample taken from Mill A, at least three samples were taken from the other mills.

The carotene profile of the fiber oil, as determined by HPLC with a binary solvent system (acetonitrile and dichloromethane), a C_{18} reverse-phase column and a variable wavelength detector, is shown in Table 3. The major carotenes present in Malaysian palm oil from the cultivated Tenara palm are α- and β-carotenes, which comprise about 90% of the total carotenes present. However, in the case of fiber oil, although the major carotenes are still α- and β-carotenes, these constitute only about 50% of the total carotenes present. Slightly higher amounts of phytoene (11.9%), lycopene (14.1%), ζ-carotene (7.6%), and δ-carotene (6.9%) are found. Other carotenes present are γ-carotene, α-zeacarotene, β-zeacarotene, neurosporene, and phytofluene.

Carotenes, a class of C_{40} polyunsaturated hydrocarbons that impart a characteristic orange-red color to palm oil, are important for human physiology. In particular, α- and β-carotenes are known for their provitamin A activities because they can be transformed into vitamin A *in vivo*. The vitamin A equivalents of α-, β-, and γ-carotenes and β-zeacarotene (which are present in palm-pressed fiber oil) are 0.9, 1.67,

TABLE 3
Composition (%) of Carotenes

	Fiber oil	Oil from Tenara palm[a]
Phytoene	11.87	1.27
Phytofluene	0.40	0.06
β-Carotene	30.95	56.02
α-Carotene	19.45	35.06
Cis-α-Carotene	1.17	2.49
ζ-Carotene[b]	7.56	0.69
δ-Carotene	6.94	0.83
γ-Carotene	2.70	0.33
Neurosporene[c]	3.38	0.29
β-Zeacarotene	0.37	0.74
α-Zeacarotene	trace	0.23
Lycopene[d]	14.13	1.30
Total (ppm)	4520–5600	500–700

[a]Commercial Malaysia crude palm oil.
[b]One *trans* and two *cis* isomers.
[c]One *trans* and one *cis* isomer.
[d]One *trans* and three *cis* isomers.

TABLE 4
Composition (%) of Tocopherol (T) and Tocotrienols (T_3) from Various Sources

Fiber oil	α-T (%)	α-T_3 (%)	γ-T_3 (%)	δ-T_3 (%)	Total T + T_3 (ppm)
$CHCl_3$ extract	67.7	18.6	15.7	trace	2300–3200
Hexane extract	61.1	15.4	18.0	5.5	2020–2640
Liquid CO_2 extract	57.0	17.4	20.5	5.1	2870–3220
Crude palm oil	22.0	20.0	46.0	12.0	600–1000

0.75, and 0.42 IU, respectively. Lycopene, which is also present, is an efficient quencher of singlet oxygen and is an effective antioxidant. Epidemiological studies in the 1980s strongly associated β-carotene with the prevention of certain types of cancers, such as oral, pharyngeal, lung, and stomach cancer (4–6). In fact, the National Institutes of Health has identified β-carotene as one of the top-ten cancer preventive agents. What is more interesting is the recent report on α-carotene, which has been shown by *in vitro* studies to be tenfold more potent as an anti-cancer agent than β-carotene (6). In addition, a recent study has also indicated that β-carotene possesses an anti-atherosclerotic effect because it is able to lower plaque levels in arteries (7).

Vitamin E. The major vitamin E compounds present in Malaysian palm oil from the Tenara palm are tocotrienols, which constitute about 70–80% of the total vitamin E present. However, in fiber oil, the major compound is α-tocopherol (57–68%), and the rest is tocotrienols (α-tocotrienol: 15–19%, γ-tocotrienol: 15–21%, and δ-tocotrienol: 1–6%). The compositions are given in Table 4.

Besides possessing vitamin E activity, tocopherols and tocotrienols are natural antioxidants. α-Tocopherol and α-tocotrienol have been reported to have anti-cancer properties in experimental animals. Among the tocotrienols, γ- and δ-isomers are the more potent anti-tumor promoters *in vitro* (8). While α-tocotrienol can suppress elevation of the cholesterol level in blood, γ-tocotrienol is able to prevent aggregation of platelets in blood as well (9).

Sterols. The fiber oil contains sterols that show a similar compositional profile to that from crude palm oil, with β-sitosterol present at 56–59%, campesterol at 19–22%, and stigmasterol at 18–20% (Table 5). Cholesterol, as expected, is present in negligible quantities. Sterols, if recovered, will have potential uses in the pharmaceutical industry for conversion into steroid derivatives. In fact, β-sitosterol is known to have the beneficial effect of being hypocholesterolemic (10).

Fatty acid composition. The residual oil extracted from fibers collected from commercial palm oil mills is from single-screw pressings and normally also contains palm kernel oil constituents, as shown by the higher percentage of C_{12} and C_{14} fatty acids (Table 6). This is not surprising because the relatively high pressure exerted during single pressing results in some broken nuts as well as kernels. This kernel debris is probably trapped and mixed with the fiber.

Quality of fiber oil. Quality parameters of fiber oil from single-pressed fibers have also been determined, and the results are: free-fatty acid (%), 7–11; peroxide value (meq/kg), 4–8; anisidine value, 9–17; phosphorus (ppm) (phosphorus content in liquid CO_2 extract is 5–10 ppm, whereas other solvent extracts are 700–1200 ppm), 5–1200; percentage of oil extracted, 4–7. Because the fiber oil contains higher free fatty acids, and other quality parameters, such as peroxide value and anisidine value, are also generally higher than those for Malaysian crude palm oil, the oil has to be refined before it can be utilized as a source for micronutrient.

In conclusion, a unique feature of palm oil is that it contains valuable minor components, principally carotenoids, vitamin E (tocopherol and tocotrienols) and sterols, which have useful nutritional and medicinal properties. These compounds, surprisingly, are present in much higher quantities in the palm-pressed fiber oil than in commercial crude palm oil. Thus, palm-pressed fiber, instead of being burned for fuel, can provide a good source of oil suitable for the recovery of carotenoids, vitamin E, and sterols.

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TABLE 5
Composition (%) of Sterols from Various Oil Sources

Sample of fiber oil	β-Sitosterol (%)	Stigmasterol (%)	Campesterol (%)	Others (%)	Total sterols (ppm)
$CHCl_3$ extract	58.4	18.7	19.9	3.0	6906–8200
Hexane extract	56.0	19.6	21.2	3.2	7050–8490
Liquid CO_2 extract	56.5	19.0	22.0	2.5	4509–5200
Fiber oil from hybrid palm[a]	67.4	14.7	14.5	3.3	6030
Crude palm oil (from mill)	57.0	15.0	24.0	4.0	250–620
Tenera palm oil[b]	62.1	18.8	16.6	2.5	783

[a]This sample is extracted by hexane. [b]Commercial crude palm oil from Malaysia.

TABLE 6
Fatty Acid Composition (%) of Oil Extracts from Palm-Pressed Fiber

Fatty acid	$CHCl_3$ extract	Hexane extract	CO_2 extract	CPO[a]	CPKO[a]
$C_{6:0}$	0.1	trace	trace	—	0.3
$C_{8:0}$	2.4	1.8	0.8	—	4.4
$C_{10:0}$	1.8	1.3	0.6	—	3.7
$C_{12:0}$	17.4	13.8	8.0	0.2	48.3
$C_{14:0}$	6.9	6.2	4.2	1.1	15.6
$C_{16:0}$	31.0	33.9	38.0	44.0	7.8
$C_{16:1}$	—	—	—	0.1	—
$C_{18:0}$	2.9	3.3	3.0	4.5	2.0
$C_{18:1}$	29.7	31.7	36.8	39.2	15.1
$C_{18:2}$	6.9	7.1	8.0	10.1	2.7
$C_{18:3}$	0.8	0.6	0.3	0.4	—
$C_{20:0}$	0.1	0.1	0.1	0.4	—

[a]CPO, Malaysian crude palm oil; CPKO, Malaysian palm kernel oil.

REFERENCES

1. Bockennoogen, H.A., *Analysis and Characterization of Oil, Fat and Fat Products,* Interscience, New York, 1974, Vol. 2, pp. 648–656.
2. Tan, B., and L. Brzuskiewicz, Separation of Tocopherol and Tocotrienol Isomer Using Normal- and Reverse-Phase Liquid Chromatography, *Anal. Biochem. 180*:368–373 (1989).
3. Peto, R., R. Doll, J.D. Buckley, and M.B. Sporn, Can Dietary Beta-Carotene Materially Reduce Human Cancer Rates, *Nature 290*:201–208 (1981).
4. Suda, D., J. Schwartz, and G. Shklar, Inhibition of Experimental Oral Carcinogenesis by Tropical Beta-Carotene, *Carcinogenesis 7*:711–715 (1986).
5. Normal, J.T., and K.B. Tapan, Does Beta-Carotene Prevent Cancer? A Critical Appraisal, *Nutr. Res. 8*:685–701 (1988).
6. Murakoshi, M., J. Takayasu, O. Kimura, E. Kohmura, H. Nishino, A. Iwashima, J. Okuzumi, T. Sakai, T. Sugimoto, J. Imanishi, and R. Isasaki, Inhibitory Effect of α-Carotene on Proliferation of Human Neuroblastoma Cell Line GOTO, *J. Natl. Cancer Inst. 81*:1649–1652 (1989).
7. Gaziano, J.M., J.E. Manson, P.M. Fidker, J.E. Buring, and H. Hennekens, Beta-Carotene Therapy for Chronic Stable Angina, paper presented at Annual Meeting of the American Heart Association, Dallas, Texas, November 1990.
8. Goh, S.H., N.H. Hew, A.W. Norhanom, and M. Yadau, Inhibition of Tumour Promotion by Various Palm Oil Tocotrienol, *Int. J. Cancer 57*:529–531 (1994).
9. Mahadevappa, V.G., F. Sicilia, and B.J. Holub, Effect of Tocotrienol Derivatives on Collagen and ADP—Induced Human Platelet A Aggregation, in *1989 PORIM International Palm Oil Development Conference,* PORIM Press, Kuala Lumpur, September 5–9, 1989, pp. 36–38.
10. Ong, A.S.H., Y.M. Choo, and C.K. Ooi, in *Developments in Palm Oil,* edited by R.J. Hamilton, Chapman & Hall Ltd., Hampshire, 1995, pp. 153–191.

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