

Quality of Residual Oil from Palm-Pressed Mesocarp Fiber (*Elaeis guineensis*) Using Supercritical CO₂ With and Without Ethanol

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ABSTRACT: The qualities of oils extracted from fresh and dried palm-pressed mesocarp fiber were evaluated. The means of extraction included conventional solvent extraction and supercritical carbon dioxide (SC-CO₂) extraction with and without addition of ethanol. Extraction efficiency using pure SC-CO₂ and the effect of moisture content on efficiency were studied. Minor components, such as vitamin E, carotenoids, squalene and phytosterols, obtained by different methods were compared. The quality of oil recovered from fresh palm-pressed fiber is generally better than that of oil recovered from dried fiber. The SC-CO₂ extraction rate was lower for fresh fiber than for dried fiber. The incorporation of ethanol with SC-CO₂ resulted in oil with higher oxidative stability than did SC-CO₂ alone. Concentrations of minor components and the acylglycerol compositions of the oils extracted from both types of fibers were similar.

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Crude palm oil (CPO) is obtained by mechanical extraction of the mesocarp and exocarp of palm fruitlets. The residue remaining after extraction, known as palm-pressed fiber, contains approximately 5–6% residual oil, including high contents of minor components such as carotenoids, tocopherols, tocotrienols, phytosterols, and squalene (1). Palm-pressed fiber represents approximately 15% of the palm fresh fruit bunch processed. In 2001, Malaysian palm oil mills produced 9.21 million metric tons of palm-pressed fiber (2). Owing to their inability to recover the residual oil in palm-pressed fiber, most palm oil mills burn the fresh fiber along with palm kernel shells for electricity generation, resulting in the loss of much valuable oil and nutrients. The application of supercritical fluid extraction to palm-pressed fiber was evaluated as a novel process to recover the fiber oil and its associated nutritional components.

In principle, solvent extraction may be used for the recovery of fiber oil from palm-pressed fiber; however, this process is economically unviable and environmentally problematic owing to the large amounts of solvent consumed. These short-

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comings can be overcome by applying supercritical carbon dioxide (SC-CO₂) extraction methods, with or without the use of minor quantities of modifiers such as alkyl alcohols or short-chain alkanes.

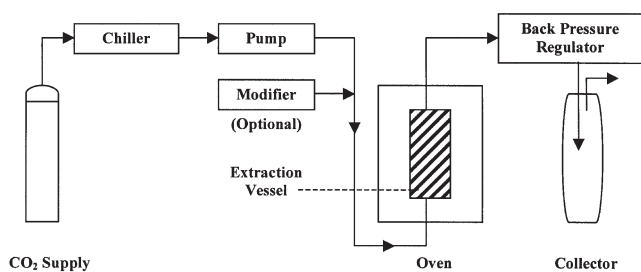
The use of SC-CO₂ extraction continues to expand into a wide field of applications including coffee decaffeination (3); extraction of essences from hops (4); extraction of oily substances from canola seed (5), bacuri shell (6), tucuma seed (7), pungent paprika (8), passion fruit (9), borage seed (10), and olive husk oil (11), as well as extraction of pigments from sweet potatoes (12). One particular advantage of SC-CO₂ extraction over conventional and existing extraction technologies is the ability to produce products free of solvent residues (13). Although the equilibrium solubility of oil in SC-CO₂ is low relative to hexane, CO₂ is often considered the preferable solvent because of its nontoxic, nonflammable, and environmentally friendly properties. The objective of the present study was to compare the qualities of fiber oils recovered by using SC-CO₂, SC-CO₂/ethanol, and *n*-hexane as extraction solvents. The compositions of the extracted oils and other selected oil quality parameters were measured.

EXPERIMENTAL PROCEDURES

Materials. Fresh palm-pressed fiber was collected from the Malaysian Palm Oil Board (MPOB) Palm Oil Mill Technology Centre (Negeri Sembilan, Malaysia). A portion of the fresh fiber was dried at 60°C for 8 h before use. Both fresh and dried fiber were flushed with nitrogen gas and stored at –5°C.

Reagents and chemicals. CP-grade CO₂ of 99.995% purity was purchased from Malaysia Oxygen Berhad (Selangor, Malaysia). Myristic acid (C14:0), palmitic acid (C16:0), stearic acid (C18:0), and oleic acid (C18:1); monopalmitin, 1,2- and 1,3-dipalmitin, and tripalmitin, with purities of 99%; β-sitosterol (95%); campesterol (98%); stigmasterol (95%); and cholesterol (99%) standards were purchased from Sigma Aldrich Inc. (St. Louis, MO). Silylating reagent N,O-bis(trimethylsilyl) trifluoroacetamide with 1% trimethylchlorosilane was purchased from Fluka Chemicals (Buchs, Switzerland). All solvents of chromatographic and analytical grade were purchased from Merck (Darmstadt, Germany).

SC-CO₂ apparatus and extraction procedure. The SC-CO₂ extraction system, Model CO-960 (JASCO, Tokyo, Japan)



SCHEME 1

complete, had a column oven (JASCO CO-960), intelligent positive displacement HPLC pump (JASCO PU-980), digital back-pressure regulator (JASCO 880-81), and extraction vessel (Thar Designs, Inc., Pittsburgh, PA). The CO₂ chiller was purchased from Polyscience (Warrington, PA). A Waters 510 HPLC co-solvent pump (Milford, MA) was used. The flow diagram of the SC-CO₂ system is shown in Scheme 1.

Fresh or dried palm-pressed fiber (25.0 g) was charged into a 100-mL extraction vessel and extracted with SC-CO₂ with and without ethanol as co-solvent. The CO₂ flow rate ranged from 3 to 10 mL/min; and when ethanol was used, it was incorporated into the SC-CO₂ stream at a flow rate of 0.5 mL/min. SC-CO₂ was pumped continuously from the top through the vertical extraction vessel. Extractions were conducted at 50 and 80°C and 30 MPa pressure. Each extraction was carried out for approximately 8 h. The oil-laden stream was depressurized by expanding through a heated needle valve to atmospheric pressure, and the fiber oil was collected in a receiver. Samples were stored at -5°C until analyzed. All extractions were conducted in triplicate and mean values reported.

Analyses. The oil samples were analyzed according to MPOB Test Methods for deterioration of bleaching index (DOBI), PV, and total carotene content (14–16). FFA, MAG, DAG, TAG, phytosterols, and squalene were determined by using a Hewlett-Packard GC-FID (5890 series II; Agilent Tech-

nologies, Palo Alto, CA) fitted with a capillary column (15 m × 0.32 mm i.d., SGE, Austin, TX) coated with 5% phenyl/95% polysilphenylene-siloxane (17). Vitamin E content was determined by HPLC-fluorescence detector (Agilent Technologies) with a silica column (5 μm; 150 mm × 4.6 mm i.d.; Zorbax, Agilent Technologies) using 1.0 mL/min of *n*-hexane/THF/isopropyl alcohol (93.9:5.7:0.4, by vol). The excitation and emission wavelengths were set at 292 and 326 nm, respectively. Oxidative stability of the oil was measured by using the Rancimat method according to AOCS Cd 12b-92 (18) (Metrohm AG, Hersau, Switzerland). All analyses were carried out in triplicate.

RESULTS AND DISCUSSION

Effect of CO₂ flow rate. The extraction efficiency of oil from dried fiber at different CO₂ flow rates was studied. Extraction curves for palm fiber oil at different flow rates are shown in Figure 1. By using SC-CO₂, 0.054 g oil per g of dried fiber was recovered. Extraction efficiency increased with increasing CO₂ flow rate; however, the magnitude of increased efficiency was not linearly proportional to the increase in CO₂ flow rate. The extraction rates of fiber oil at 3.0, 5.0, and 10.0 mL/min of CO₂ flow were 8.0, 26.7, and 40.0 mg/min, respectively. Extraction times for 95% recovery at 3.0, 5.0, and 10.0 mL/min of CO₂ flow were about 400, 200, and 100 min, respectively. Total CO₂-extractable oil recovery was 93.1% relative to *n*-hexane. Although the penetrating power of pure SC-CO₂ is stronger than solvent (e.g., *n*-hexane) at supercritical conditions owing to higher diffusivity, the solubility of polar lipids is higher in *n*-hexane, resulting in 6–7% more yield of fiber oil compared with SC-CO₂ extraction.

Effect of fiber condition. As a result of the high moisture content of palm fruits, sterilization of the palm fruits is necessary to deactivate the lipases and prevent the enzymatic hydrolysis of lipids to FFA. Residual moisture levels in fresh fiber

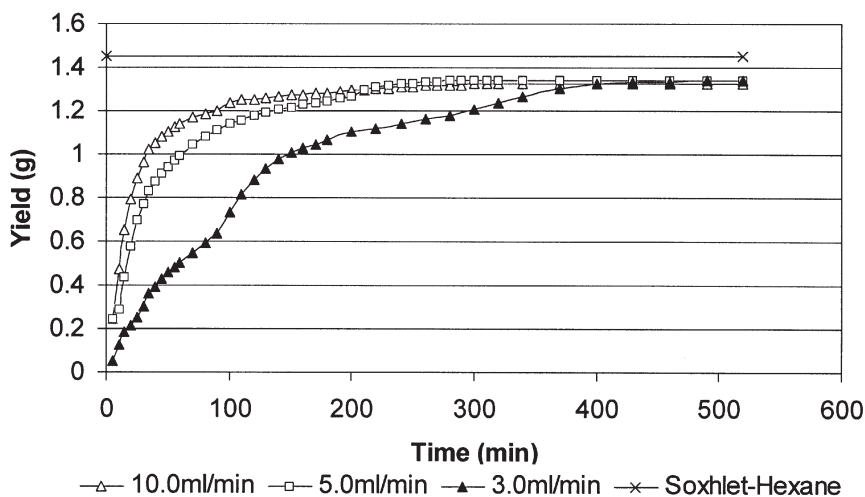


FIG. 1. Effect of CO₂ flow rate on the extraction of residual oil from dried palm-pressed fiber using supercritical CO₂ (SC-CO₂) and *n*-hexane.

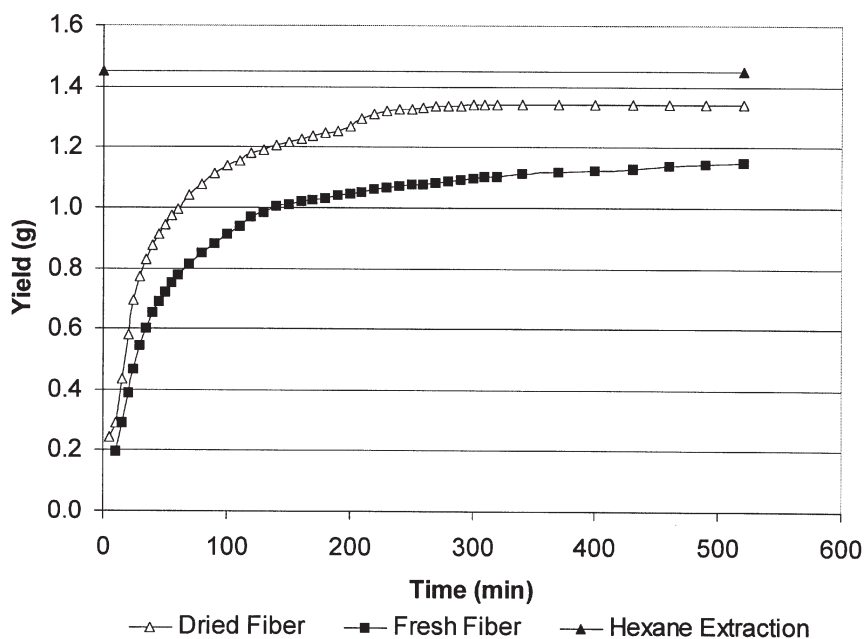


FIG. 2. Effect of moisture content in palm-pressed fiber on the extraction of residual oil using SC-CO₂ at 50°C and 30 MPa. For abbreviation see Figure 1.

ranged between 35 and 40%, depending on the pressure using in mechanical pressing. The presence of moisture alters the phase characteristics of SC-CO₂, reduces extraction rates, and affects the quality of the extracted fiber oil. The extraction rate of fiber oil was 25 mg/min from fresh fiber and 40 mg/min from the dried fiber (Fig. 2). The extraction rate of fresh fiber increased as water was removed. Polar lipids, such as phospholipids and glycolipids, were recovered early in the extraction process because of the presence of water and the resulting polarity of the extraction solvent system.

Quality of palm-pressed fiber oil. The qualities of the oils extracted from fresh and dried fibers using SC-CO₂ and *n*-hexane are reported in Table 1. Oven-drying of the fiber had a detrimental effect on oil quality, resulting in increased PV. The PV of fiber oils recovered from dried fiber ranged from 1.83 to 2.34

mequiv/kg compared with 0.46–0.84 mequiv/kg for fiber oil recovered from fresh fiber.

Fiber oil extracted with *n*-hexane was the most oxidatively stable of the recovered fiber oils, as determined by the accelerated oxidative stability method. Induction periods of more than 48 and 33.7 h were observed for *n*-hexane-extracted fiber oil extracted from fresh and dried fibers, respectively. Similar results have been reported for sunflower oils (19). The superior oxidative stability of hexane-extracted oil could be explained by the high phosphorus content compared with SC-CO₂ extracted oil (20). This observation is supported by the hypothesis that phospholipids may reduce the rate of air penetration into the sample by acting as an oxygen barrier at the oil/air interface, and thus prolong the oxidative stability of the oil.

Oxidative stability of fiber oil obtained from SC-CO₂-ex-

TABLE 1
Quality Parameters of Palm-Pressed Fiber Oil^a

Extraction method	Extraction conditions	PV (mequiv O ₂ /kg)	Oxidative stability (h)	DOBI
Fresh fiber				
SC-CO ₂	50°C, 30 MPa	0.46 ± 0.03	21.9 ± 0.15	2.21 ± 0.12
SC-CO ₂	80°C, 30 MPa	0.52 ± 0.03	18.8 ± 0.11	2.19 ± 0.09
SC-CO ₂ /EtOH	50°C, 30 MPa	0.51 ± 0.04	29.4 ± 0.19	2.13 ± 0.11
<i>n</i> -Hexane	76–80°C	0.84 ± 0.04	>48	2.01 ± 0.08
Dried fiber				
SC-CO ₂	50°C, 30 MPa	1.83 ± 0.05	16.6 ± 0.32	2.69 ± 0.05
SC-CO ₂	80°C, 30 MPa	2.34 ± 0.06	16.1 ± 0.22	2.58 ± 0.10
<i>n</i> -Hexane	76–80°C	2.23 ± 0.04	33.7 ± 0.34	2.08 ± 0.07

^aSC-CO₂, supercritical CO₂; DOBI, deterioration of bleachability index. Data reported are the means of three replicate analyses of independent samples.

TABLE 2
Composition of Acylglycerols and FFA in Palm-Pressed Fiber Oil^a (%)

Extraction method	Extraction condition	FFA	MAG	DAG	TAG
Fresh fiber					
SC-CO ₂	50°C, 30 MPa	3.46 ± 0.05	0.35 ± 0.02	0.85 ± 0.02	94.30 ± 1.03
SC-CO ₂	80°C, 30 MPa	3.84 ± 0.04	0.38 ± 0.03	0.89 ± 0.05	93.73 ± 0.79
SC-CO ₂ /EtOH	50°C, 30 MPa	3.88 ± 0.04	0.41 ± 0.03	0.96 ± 0.03	93.63 ± 0.83
<i>n</i> -Hexane	76–80°C	3.94 ± 0.03	0.37 ± 0.04	1.14 ± 0.03	93.52 ± 1.12
Dried fiber					
SC-CO ₂	50°C, 30 MPa	3.79 ± 0.05	0.31 ± 0.02	0.86 ± 0.04	93.73 ± 0.32
SC-CO ₂	80°C, 30 MPa	3.78 ± 0.06	0.28 ± 0.03	0.83 ± 0.04	93.78 ± 0.77
<i>n</i> -Hexane	76–80°C	3.70 ± 0.04	0.27 ± 0.02	0.92 ± 0.02	93.87 ± 0.62

^aFor abbreviation see Table 1. Data reported are the means of three replicate analyses of independent samples.

tracted fresh fiber (18.8–29.4 h) was better than that for oil recovered from SC-CO₂-extracted dried fiber (16.1–16.6 h). One reason for improved stability of fiber oil from fresh fiber may be the higher contents of phospholipids. The presence of water in fresh fiber will polarize the SC-CO₂ stream, increasing the tendency to extract more polar compounds such as phospholipids. Choo *et al.* (21) reported that the content of phospholipids in palm-pressed fiber oil can be as high as 4.68%. Phospholipids have synergistic effects with α -tocopherol and contribute to the stability of the fiber oil (22). Lau *et al.* (23) also discovered the presence of water-soluble phenolic compounds with antioxidant activity in residual oil extracted from fresh palm-pressed fiber. The amount of water-soluble phenolic compounds was determined in a previous study (23) and will not be discussed here. The extra 6–7% yield of polar lipids recovered by *n*-hexane was mainly phospholipids and, we believe, led to the superior oxidative stability over SC-CO₂-extracted fiber oil.

By using ethanol as a co-solvent, the polarity and solvating

power of SC-CO₂ were significantly increased, which enabled more phospholipids and glycolipids to be extracted. The oil extracted from fresh fiber using SC-CO₂/ethanol was more stable than the 100% SC-CO₂-extracted fiber oil, with induction periods of 29.4 and 18.8 h, respectively.

DOBI is an indicator of the ease of bleaching and degumming of CPO; the higher the DOBI, the higher the quality of the CPO. Higher DOBI oils tend to be cheaper to refine, as a cost savings results from reduced dosages of phosphoric acid and bleaching earth. It was observed that oil obtained from SC-CO₂-extracted dried fiber had higher DOBI values than oil extracted from fresh fiber. The low DOBI value was attributable to increased UV absorbance at 269 nm, the wavelength where phospholipids absorb. Although the phospholipids present in fiber oil help stabilize oil against oxidation, their presence also increases the refining cost. It is possible to recover phospholipids in higher purity if palm-pressed fiber is first de-oiled by SC-CO₂, followed by extraction of the polar compounds with SC-CO₂/ethanol.

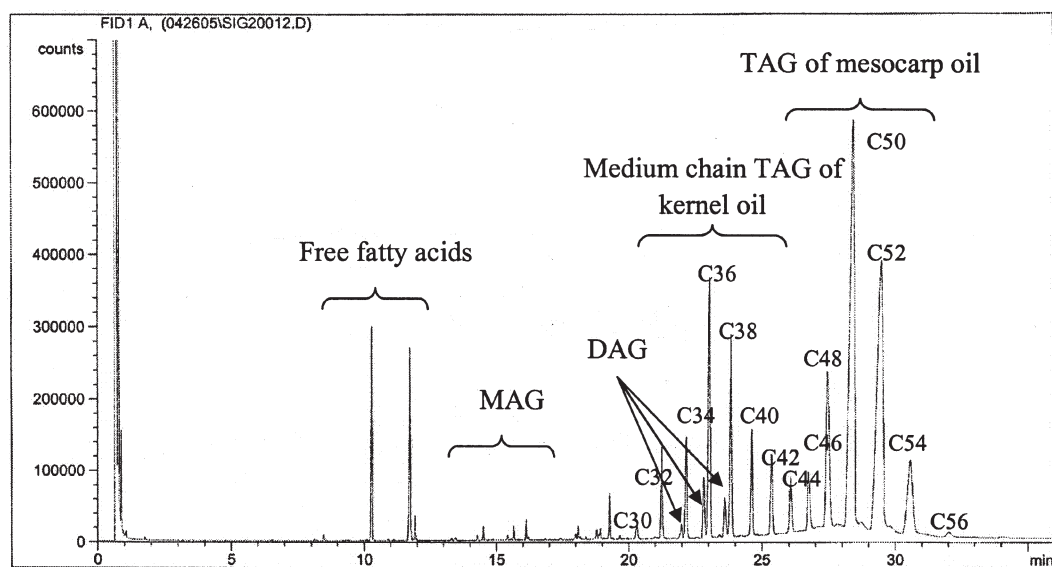


FIG. 3. GC-FID chromatogram of palm-pressed fiber oil.

TABLE 3
Concentrations of Minor Components in Palm-Pressed Fiber Oil^a (mg/kg)

Extraction method	Extraction condition	Squalene	Phytosterols	Carotenes	Vitamin E
Fresh fiber					
SC-CO ₂	50°C, 30 MPa	1102 ± 21	4429 ± 31	2909 ± 15	1979 ± 14
SC-CO ₂	80°C, 30 MPa	1321 ± 18	4409 ± 27	3424 ± 13	2372 ± 14
SC-CO ₂ /EtOH	50°C, 30 MPa	1343 ± 17	3007 ± 24	3487 ± 20	2367 ± 11
<i>n</i> -Hexane	76–80°C	1117 ± 20	4349 ± 33	2933 ± 11	1981 ± 18
Dried fiber					
SC-CO ₂	50°C, 30 MPa	1642 ± 22	4749 ± 31	4424 ± 16	2303 ± 19
SC-CO ₂	80°C, 30 MPa	1633 ± 12	4568 ± 19	4638 ± 21	2584 ± 17
<i>n</i> -Hexane	76–80°C	1495 ± 25	4610 ± 22	4007 ± 15	2251 ± 13

^aFor abbreviation see Table 1. Data reported are the means of three replicate analyses of independent samples.

The compositions of acylglycerols and FFA are shown in Table 2. FFA levels ranged from 3.70 to 3.94%, MAG from 0.27 to 0.41%, DAG from 0.83 to 1.14%, and TAG from 93.52 to 94.30%. The source of the 31.5% medium-chain TAG was palm kernel oil, as some broken kernels may be trapped in the fiber matrix during screw pressing of CPO. A typical GC-FID chromatogram of palm-pressed fiber oil is given in Figure 2.

Minor components. Concentrations of squalene, phytosterols, carotenoids, and vitamin E in fiber oils ranged from 0.11 to 0.16%, 0.30 to 0.47%, 0.29 to 0.46%, and 0.20 to 0.26%, respectively (Table 3).

(i) **Vitamin E.** The concentrations of vitamin E extracted from fresh and dried palm-pressed fiber by using SC-CO₂, SC-CO₂/ethanol, and hexane ranged from 2000 to 2600 mg/kg (Table 3), which is twofold higher than CPO, with concentrations of 600–1000 mg/kg (1). The vitamin E profile of fiber oil (Table 4) was very different from CPO, with, on average, 63.7% α -tocopherol, 15.8% α -tocotrienol, 18.3% γ -tocotrienol, and 2.2% δ -tocotrienol compared with 22% α -tocopherol, 20% α -tocotrienol, 46% γ -tocotrienol, and 12% δ -tocotrienol in CPO (1).

(ii) **Carotenoids.** The concentrations of carotenoids in oil extracted from dried fiber using SC-CO₂ and hexane were higher (4000–4600 mg/kg) than from fresh fiber (2900–3500 mg/kg) (Table 3). In SC-CO₂ extraction, the later fractions had an intense reddish color, indicating that carotenoids from TG

can readily be fractionated. The concentration of lycopene (14.1%), one of the carotenoid compounds, was 10 times higher in fiber oil than in CPO (1). Lycopene is particularly effective at quenching the destructive potential of singlet oxygen (24).

(iii) **Phytosterols.** The phytosterol profile, shown in Table 5, is 70.1% β -sitosterol, 18.8% stigmasterol, 8.2% campesterol, and 2.9% cholesterol. The total concentration of phytosterols (Table 3) is almost 10 times higher in fiber oil than in CPO, with concentrations in the latter ranging from 250 to 650 mg/kg (1). Thus, fiber oil may be considered a rich source of phytosterols, which can easily be recovered from readily available palm-pressed fiber.

(iv) **Squalene.** The concentration of squalene in fiber oil has not been reported previously. We determined that the concentration of squalene in fiber oil extracted from dried fiber ranged from 1500 to 1600 mg/kg (Table 3), which is higher than that recovered from fresh fiber and commercial CPO, with concentrations of 1100–1300 and 250–500 mg/kg, respectively (25). Fractionation of squalene from lampante olive oil using SC-CO₂ has been successfully demonstrated by Bondioli *et al.* (26). Owing to the high concentration of squalene in palm fiber oil, countercurrent SC-CO₂ extraction potentially can be applied to obtain fractions enriched with squalene.

SC-CO₂ extraction of palm-pressed fiber has the potential to be used for recovery of fiber oil and its indigenous value-added

TABLE 4
Composition of Vitamin E in Palm-Pressed Fiber Oil^a (%)

Extraction method	Extraction condition	α -T	α -T3	γ -T3	δ -T3
Fresh fiber					
SC-CO ₂	50°C, 30 MPa	59.0 ± 1.3	18.3 ± 1.0	19.7 ± 1.2	3.0 ± 0.2
SC-CO ₂	80°C, 30 MPa	63.0 ± 2.1	14.3 ± 1.1	20.8 ± 1.5	1.9 ± 0.1
SC-CO ₂ /EtOH	50°C, 30 MPa	62.5 ± 1.7	17.4 ± 0.9	18.3 ± 1.2	1.8 ± 0.2
<i>n</i> -Hexane	76–80°C	62.9 ± 1.2	14.2 ± 1.5	20.9 ± 0.9	2.0 ± 0.2
Dried fiber					
SC-CO ₂	50°C, 30 MPa	64.5 ± 1.4	16.5 ± 1.2	16.3 ± 1.3	2.6 ± 0.2
SC-CO ₂	80°C, 30 MPa	66.0 ± 1.1	16.5 ± 1.4	15.4 ± 1.0	2.1 ± 0.2
<i>n</i> -Hexane	76–80°C	68.2 ± 1.7	13.3 ± 1.0	16.6 ± 1.1	1.9 ± 0.1

^a α -T, α -tocopherol; α -T3, α -tocotrienol; γ -T, γ -tocopherol; δ -T3, δ -tocotrienol; for other abbreviation see Table 1. Data reported are the means of three replicate analyses of independent samples.

TABLE 5
Composition of Phytosterols in Palm-Pressed Fiber Oil^a (%)

Extraction method	Extraction condition	Cholesterol	Campesterol	Stigmasterol	β -Sitosterol
Fresh fiber					
SC-CO ₂	50°C, 30 MPa	2.6 ± 0.1	8.2 ± 0.1	19.0 ± 0.2	70.1 ± 0.4
SC-CO ₂	80°C, 30 MPa	2.6 ± 0.2	9.0 ± 0.3	19.1 ± 0.3	69.3 ± 0.3
SC-CO ₂ /EtOH	50°C, 30 MPa	2.3 ± 0.1	8.6 ± 0.2	18.8 ± 0.1	70.3 ± 0.3
<i>n</i> -Hexane	76–80°C	3.9 ± 0.2	7.7 ± 0.2	18.3 ± 0.2	70.1 ± 0.2
Dried fiber					
SC-CO ₂	50°C, 30 MPa	2.8 ± 0.1	7.5 ± 0.3	18.6 ± 0.3	71.1 ± 0.3
SC-CO ₂	80°C, 30 MPa	3.2 ± 0.2	9.1 ± 0.1	19.3 ± 0.2	68.4 ± 0.2
<i>n</i> -Hexane	76–80°C	2.7 ± 0.2	7.2 ± 0.2	18.5 ± 0.1	71.6 ± 0.3

^aFor abbreviation see Table 1. Data reported are the means of three replicate analyses of independent samples.

minor components. The palm fiber oil extracted by SC-CO₂ was cleaner in terms of better transparency (data not shown) as compared with *n*-hexane and SC-CO₂/ethanol extracted fiber oil.

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