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Supercritical Fluid Extraction of Palm Kernel Oil from Palm Kernel Cake

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ABSTRACT

Supercritical fluid carbon dioxide (SC-CO₂) at pressure 19.8 MPa and temperature 51°C with different amount of ethanol (0-100 mL) was studied the extraction of palm kernel oil from palm kernel cake. The amount of oil produced from SFE and Modified ethanol-CO₂ are proportional to the amount of ethanol. It was found that α -tocopherol, α -tocotrienol, sterols and fatty acid such lauric acid, myristic acid and oleic acid were present in all of the palm kernel oil sample.

Key words: Palm kernel, supercritical fluid extraction, vitamin E, fatty acid

INTRODUCTION

Supercritical Fluid Extraction (SFE) is the process of separating extractant from another matrix by using supercritical fluid as extracting solvent. Carbon dioxide (CO₂) is the most widely supercritical fluid and sometimes modified by co-solvents such as ethanol or methanol. Extracting conditions for supercritical CO₂ are above the critical temperature of 31°C and critical pressure of 74 bar (Yin *et al.*, 2005; Birtigh *et al.*, 1995).

Vitamin E represents a family of eight structurally related compounds (four tocopherols and four tocotrienols) that occur naturally as components of palm oils. The isomer with the highest vitamin E activity is α -tocopherol and this is an important additive to all kinds of food products. The naturally occurring tocopherols and tocotrienols constitute the majority of the vitamin E group of components and they are composed of eight vitamers α , β , γ and δ tocopherol and their four corresponding unsaturated congeners, namely α , β , γ and δ tocotrienol (Hasselwander *et al.*, 2002).

Palm Kernel Cake (PKC) is rich in lauric acid, C12 (48.3%) and other major fatty acids such as myristic, C14 (15.6%) and oleic acid C18:1 (15.1%) (Zaidul *et al.*, 2006, 2007). Palm kernel oil also contains other fatty acid but in a small amount such palmitic C16, capric C10, caprylic C8, stearic C18 and linoleic C18 and has some unknown fatty acid.

PKC also referred as Palm Kernel Expeller (PKE), has long been known to be an important ingredient for the formulation of animal feeds (Chu *et al.*, 2002). PKC is obtained from palm nut. It contains about 45-50% oil.

MATERIALS AND METHODS

Collection of raw palm kernel cake: Palm kernel cake (*E. guineensis*) was supplied by Lumadan Mill, Borneo Samudera Sdn. Bhd., Beaufort, Sabah.

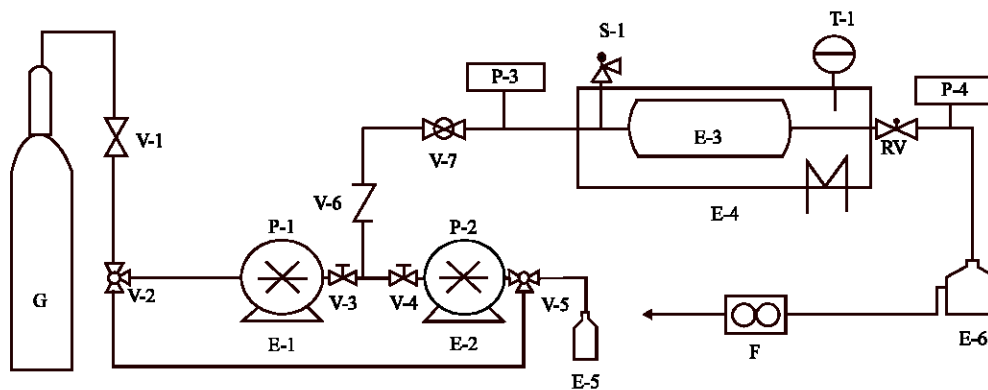


Fig. 1: Experimental apparatus. E-1, E-2: Pumps; E-3: Extraction vessel; E-4: Thermostated bath; E-5: Co-solvent vessel; E-6: Separation vessel; F: Flowmeter; G: CO₂ cylinder; P: Pressure transducers; RV: Regulating valve; S-1: Safety valve; T-1: Temperature controller; V: Valves

Preparation of palm kernel samples: The PKC was washed with distilled water and then dried at temperature of 40°C for 3 days. Then it was separated into particle size range 1-2 mm by sieving. This powder was then stored in airtight glass containers.

Supercritical carbon dioxide (SC-CO₂) extraction: Liquid carbon dioxide was pumped into the heated extraction cell loaded with approximately 100 g of ground palm kernel cake sample of 1 mm to 2 mm diameter in size. Pressure was set at 19.8 MPa and temperature of 51°C. Static extraction was performed for 5 min before the pump was turned on to stabilize the temperature and another 5 min after the set pressure was reached before the exit valve was opened. The extract was collected about 1 g of oil for each fraction by varying the CO₂ flow maintained at the desired pressure and temperature of the extractor. The volume of CO₂ passed through the extraction cell was recorded at atmospheric pressure and temperature was measured with using a wet gas meter. The experimental setup is shown in Fig. 1.

Supercritical extraction using co-solvent ethanol: Experiments with cosolvent ethanol (100, 50, 45, 0 mL) were carried out at 51°C and 19.8 MP. A second pump was used to introduce ethanol. In order to make sure the CO₂ and ethanol mixture formed is a supercritical fluid, the conditions of temperature and pressure for SFE using the co-solvent were chosen taking into account the experimental critical locus of CO₂ and ethanol mixtures. The critical parameters for a CO₂ and ethanol mixture are T_c = 42°C and P_c = 8.6 MPa. Therefore, CO₂ modified with 100, 50 and 45 mL ethanol at 51°C and 19.8 MPa is a supercritical fluid as required.

Quantitative analysis on fatty acid constituents

Methyl esterification of fatty acids: Oil sample were heated to 70°C and homogenized thoroughly. A 100 µL of a test sample was mixed with 1 mL n-hexane in a 2 mL vial. A 1 µL aliquot of sodium methoxide was added to the vial which was mixed vigorously with a vortex mixer. The mixture first became clear and then turbid as sodium glyceroxide was precipitated. After a few minutes, the clear upper layer of the methyl ester was pipette off and injected into the gas chromatography (GC) using external standard methods (PORIM Test Method No. p3, 4) (PORIM, 1995).

Preparation of standard of fatty acids: Standard of the fatty acid methyl ester of lauric C12 (24.6%), myristic C14 (9.84%) and oleic C18:1 (9.84%).

Fatty acid composition analysis by gas chromatography (GC): The methyl esterified fatty acids above were analyzed for fatty acid profile using gas chromatography. A gas chromatography was used to determine the fatty acids profile. A 1 μL sample of the fatty acid methyl ester standard mixture was injected. After elution, a 1 μL of the prepared solution (clear upper layer) was injected separately onto the column. The oven temperature was set at 90°C, the detector and injector temperature was set at 250°C (isothermal condition). Helium was used as the carrier gas at the flow rate of 1 mL min^{-1} with the split ratio of 1:100, Auto Injector and TC-Wax column (length 30 $\text{m} \times 0.25$ mm internal diameter 0.25 μm and max temperature was 250, prog 260) were used (PORIM, 1995).

Qualitative and quantitative analysis of vitamin E and sterol constituents

Internal standard solution: Dissolve 0.20 g of oil sample in hexane and dilute to 100.0 mL with the same solvent.

Reference solution: Dissolve 0.100 g of sterol, α -tocopherol, α -tocotrienol and sterol in the internal standard solution and dilute to 50.0 mL with the internal standard solution in different conical flask.

Determination of tocopherols and tocotrienols and sterol: Analysis of tocopherol, tocotrienol and sterol was performed with gas chromatography system consisted of an Agilent Technologies gas chromatography equipped with an auto sampler, an Agilent technologies 7683 Series column injector and a flame ionization detector (Agilent Technologies, Palo Alto, CA, USA). Ramped oven temperature was used (from 180°C increased to 260°C at 8°C min^{-1} , increased to 280°C at 2°C min^{-1} and held for 13 min). Inlet temperature was 290°C and the detector temperature was 300°C. Helium was the carrier gas at constant flow of 1.2 mL min^{-1} . Detector (FID) air, H_2 and make-up gas (He) flows were 136, 35 and 45 mL min^{-1} . The identification of sterol, α -tocopherol, α -tocotrienol was confirmed by using the peaks of tocopherols and tocotrienols and were determined based on the retention time of standards (Malaysian Palm Oil Board, Malaysia). And the amount of sterol, α -tocopherol, α -tocotrienol were calculated using internal standard.

RESULTS AND DISCUSSION

The results of quantitative analysis of extraction for different amounts of ethanol are listed in Table 1. While the result on qualitative and quantitative of vitamin E, Sterol and fatty acid are tabulated in Table 2 and 3. The quantitative amount of fatty acid is in %, while for sterol and vitamin E are in ppm.

Oil and components: From Table 1, it can be seen that SFE with 100 mL ethanol co-solvent has a highest quantity of oil produced from the extraction with 3.668 g followed by modified with 50 mL ethanol, 45 mL ethanol. And the lowest amount of oil produced by extraction using pure CO_2 as a solvent.

Qualitative analysis: Qualitative analysis carried out on palm kernel oil that extracted using supercritical fluid extraction with various amounts of ethanol and shows the presence of fatty acids,

Table 1: Amount of oil extracted with different ethanol quantity

Sample	Pure CO ₂	45 mL ethanol	50 mL ethanol	100 mL ethanol
Weight before (g)	62.736	62.758	61.770	61.662
Weight after (g)	62.998	63.350	63.601	65.330
Weight of oil content (g)	0.262	0.592	1.831	3.668

Table 2: Qualitative analysis on fatty acid, vitamin E and sterol

Sample	Fatty acid			Vitamin E				Sterol
	Lauric	Myristic	Oleic	α -tocopherol	β -tocopherol	α -tocotrienol	β -tocotrienol	
Pure CO ₂	+	+	+	+	-	+	-	+
45 mL ethanol	+	+	+	+	-	+	-	+
50 mL ethanol	+	+	+	+	-	+	-	+
100 mL ethanol	+	+	+	+	-	+	-	+

Absence of component: (-); Presence of component: (+)

Table 3: Quantitative analysis on fatty acid, vitamin E and sterols

Sample	Fatty acid (%)			Vitamin E (ppm)		Sterol (ppm)
	Lauric	Myristic	Oleic	α -tocopherol	α -tocotrienol	
Pure CO ₂	59.3	21.1	19.5	230.0	300.0	650.0
45 mL ethanol	59.4	21.1	19.5	229.2	302.0	660.4
50 mL ethanol	59.5	21.2	19.3	228.0	300.6	677.2
100 mL ethanol	59.5	21.0	19.5	233.3	309.7	678.0

vitamin E and sterol (Table 2). Table 2 shows that lauric acid, myristic acid and oleic acid were present as a fatty acid in palm kernel oil. Alpha-tocopherol and alpha-tocotrienol also presence in palm kernel oil sample while both beta-tocopherol and tocotrienol are absence. Sterol is present in all palm kernel oil sample for various amount of ethanol samples.

From the Table 3, palm kernel sample with 100 mL ethanol has the highest amount of fatty acid, vitamin E and sterols. The highest amount of sterol produced is proportional with highest amount of modified ethanol. The lauric acid percentage in palm kernel oil with 100 mL ethanol and 50 mL ethanol have the higher content with 59.5%, while sample for pure CO₂ and with 45 mL ethanol have almost similar quantities of lauric acid. In the case of oleic acid also similar percentage were obtained in pure CO₂, 45 mL ethanol and 100 mL ethanol with 19.5% of oleic acid, while for 50 mL ethanol have the lower amount of oleic acid presence. Quantitative analysis of vitamin E shows that the highest amount of α -tocopherol presence is 233.3 ppm followed by 228.0, 229.2 and 230.0 ppm. The α -tocotrienol with 100 mL ethanol has the highest yield with 309.7 ppm of α -tocotrienol and followed by 306.0, 302 and 300 ppm with 50 mL ethanol, 45 mL ethanol and pure CO₂ solvent.

CONCLUSION

The presence of vitamin E, Sterols and Fatty acids in palm kernel oil was found. Supercritical CO₂ both pure and with ethanol is shown to be effective in the extraction of oil from palm kernel. For the same temperature, pressure and solvent ratio, the highest initial yield is obtained at 51°C and 19.8 MPa, with CO₂ modified with 100 mL 98% ethanol. From results, the quantity of oil produced with ethanol are higher than from pure CO₂ and the weight of oil produced also

increasing proportionally when the amount of modified increasing. Vitamin E that appeared in palm kernel oil contents, are alpha-tocopherol and alpha-tocotrienol types. And the total of vitamin E in palm kernel oil is 650 ppm compared to other conventionally method.

REFERENCES

- Birtigh, A., M. Johannsen, G. Brunner and N. Nair, 1995. Supercritical-fluid extraction of oil-palm components. *The J. Supercritical Fluids*, 8: 46-50.
- Chu, B.S., H.M. Ghazali, O.M. Lai, Y.B. Che Man and S. Yusof, 2002. Physical and chemical properties of a lipase-transesterified palm stearin/palm kernel olein blend and its isopropanol-solid and high melting triacylglycerol fractions. *Food Chem.*, 76: 155-164.
- Hasselwander, O., K. Kramer, P.P. Hoppe, U. Oberfrank and K. Baldenius *et al.*, 2002. Effects of feeding various tocotrienol sources on plasma lipids and aortic atherosclerotic lesions in cholesterol-fed rabbits. *Food Res. Int.*, 35: 245-251.
- PORIM, 1995. PORIM test methods. Palm Oil Research Institute of Malaysia, Ministry of Primary Industries, Malaysia.
- Yin, J.Z., A.Q. Wang, W. Wei, Y. Liu, W.H. Shi, 2005. Analysis of the operation conditions for supercritical fluid extraction of seed oil. *Separat. Purificat. Technol.*, 43: 163-167.
- Zaidul, I.S.M, N.A. Nik Norulaini, A.K. Mohd Omar and R.L. Smith Jr., 2006. Supercritical carbon dioxide (SC-CO₂) extraction of palm kernel oil from palm kernel as cocoa butter replacers blend. *J. Food Engin.*, 73: 210-216.
- Zaidul, I.S.M., N.A. Nik Norulaini, A.K. Mohd Omar and R.L. Smith Jr., 2007. Supercritical carbon dioxide (SC-CO₂) extraction of palm kernel oil from palm kernel. *J. Food Engin.*, 79: 1007-1014.