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Oil Extraction from Sheanut Kernel (*Vitellaria paradoxa* Gaertn) and Canarium Pulp (*Canarium schweinfurthii* Engl.) Using Supercritical Co_2 and Hexane: A Comparative Study

[1]Gilles Bernard Nkouam, [1]César Kapseu, [2]Danielle Barth, [2]Michel Dirand and [3]Jean Bosco Tchatchueng
[1]Department of Process Engineering, ENSAI-University of Ngaoundere,
P.O.Box 455 Ngaoundere, Cameroon
[2]LTMP, ENSIC-INPL, B.P. 20451 1, rue Grandville, 54001 Nancy Cedex, France
[3]Department of Applied Chemistry, ENSAI-University of Ngaoundere,
P.O.Box 455 Ngaoundere, Cameroon

Abstract: Oil extraction using CO_2 from sheanut kernel (*Vitellaria paradoxa* Gaertn.) and *Canarium* pulp (*Canarium Schweinfurthii* Engl.) has been studied in comparison with hexane extraction. Samples that were stored at 18°C and -33°C, were analyzed after 1 month, 1 and 2 years. Results showed that the extraction yields varied (dry basis) from 17.43-39.57% for shea butter and from 16.10-40.45% for *Canarium* oil. Lipids extracted with CO_2 gave highest acid values, but lowest iodine values what irrespective of the storage temperature. Shea butter analysis by gaseous phase chromatography indicated a composition in stearic and oleic acids that did not vary significantly. Butter extracted with supercritical CO_2 from sheanut kernel stored at 18°C were rich in polyunsaturated fatty acid. These results suggest that greater investigations on supercritical CO_2 extraction of these fatty materials should be encouraged because of its selectivity.

Key words: *Vitellaria paradoxa* gaertn, *Canarium schweinfurthii* Engl, supercritical carbon dioxide, extraction, lipids

INTRODUCTION

Shea (*Vitellaria paradoxa* Gaertn.) and aele tree (*Canarium schweinfurthii* Engl) are 2 non conventional lipid sources belonging to the Sapotaceae and Burseraceae families respectively. Shea is a species of the soudano-Guinean climate zone (Kassamba, 1997) while *Canarium* is a plant of the equatorial forests of tropical Africa (Aubreville, 1959). Fatty materials of sheanut kernel and the pulp of *Canarium* are susceptible to deterioration (Cheftel and Cheftel, 1984; MSDA, 2005) due to their high content of unsaturated fatty acids (Kapseu *et al*., 2001). The period of production of the shea coincides with the rainy season, where farm work by rural women is at its peak. This limits the valorisation of the shea fruit by extraction of its butter as a secondary activity for women to whom it brings a little income. In general, a larger portion of the fruits have to be stored for oil extraction later. To avoid losses during the production period of these fruits, it is indispensable to transform them (extraction of lipids) or to store them in conditions that extend their duration of utilization. Traditionally, local populations extract butter using water as solvent (SNV, 1991). Some toxic organic solvents have been used such as methanol (Maranz *et al*., 2003), chloroform (Womeni *et al*., 2002), petroleum ether (Ajiwe *et al*., 1998) and hxane (Agbo and Chatigre, 1996; Kapseu *et al*., 1999; Tchiegang *et al*., 2003). To the best of our knowledge, no study has yet been carried out on the supercritical CO_2 extraction of lipids of the sheanut kernel and the pulp of *Canarium*, a solvent which is known to be inert.

The objective of this study is to compare supercritical CO_2 and hexane extraction of the fatty materials of the sheanut kernel and the *Canarium* pulp in terms of rate of extraction yields and the properties of oils extracted.

MATERIALS AND METHODS

Biological samples: *Vitellaria paradoxa* Gaertn. (karité) and *Canarium Schweinfurthii* Engl. fruits were bought from Bambi village, a suburb of Ngaoundere in the Adamawa province of Cameroon and from Bangou village in the West province of Cameroon, respectively. Bangou village is a region with altitude of 1600 m and, is located at latitude 5°9'N and longitude 10°32'E while Bambi village is located at latitude 7°32'N and longitude 1333°78'E on

Corresponding Author: Nkouam Gilles Bernard, ENSIC-INPL Laboratoire de Thermodynamique des Milieux Polyphasés (LTMP), B.P n°451, 1, rue Grandville, F-54001 NANCY Cedex, France

Table 1: Extraction parameters for the different experiments

						Pressure (bars)			
Sam	T_{stock} (°C)	Mc (g)	Q (g)	Qm (g h^{-1})	TE (min)	P_0	P_1	P_2	P_3
Shea	18°C	8.82-10.13	85-95	831-917	135-165	220	40	30	20
	-33°C	9.02-10.06	86-105	749-994	150-180	220	40	30	20
Canarium	18°C	10.01-10.15	77-97	690-1098	120-180	220	40	30	20
	-33°C	10.01-10.09	94-117	637-1107	135-180	220	40	30	20

Sam: Sample; T_{stock}: storage Temperature in°C; Mc: Charge Mass; Qm: CO_2 mean rate; TE: Extraction time; Q: CO_2 Mass in contact with charge; (P_0, T_0): Pressure and Temperature of extractor; ($P_{1, 2, 3}$): Pressures of separators 1, 2 and 3, respectively

an altitude of 1381.66 m. After dehulling shea fruits, the nuts and the *Canarium* fruits were then stored for 1, 12 et 24 months in different conditions: at wrapped in a white polyethylene low density sachet and stored in the ambient air (temperature: 18±2°C; relative humidity: 38±6%); wrapped in the white polyethylene low density sachet and stored in a freezer of mark *Vestfrost* (temperature: -33±1°C; relative humidity: 37±6%). A thermohygrometer of mark *NOVO* was used to measure the temperature and relative humidity.

Lipids extraction: After dehulling the stored sheanuts, kernels were cut transversely in smaller pieces to ease drying. The stored *Canarium* fruits were longitudinally dehulled. The cut up samples were then dried for 48 h at 50°C using a convection oven of mark *R. Chaix, Meca* (France). After drying, samples were ground for about 20 sec using a household grinder of mark *Moulinex*. Before lipid extraction, granulometric distribution of ground samples was done using a laser granulometer (*Malvern Instruments*, England) which gave mean sizes of 555.71 and 258.95 μm for sheanut kernel and *Canarium* pulp, respectively.

Supercritical CO_2 extraction: The supercritical dynamic extraction installation is constituted of an extractor (cylinder in rustproof steel) brought up in set with three separators of *cyclone* type. It is bound to a membrane pump, of mark *Dosapro Milton Roy-Milroyal D*, which is adapted to easily attain the extraction pressure. A Coriolis force flowmeter, of mark *Microphone Motion* measures the flow rate during experimentation.

The extraction consists of putting the product in contact with the supercritical CO_2 (*Messer*, France). The supercritical CO_2 binds itself to the material due to its high affinity for the oil (30-45 min). The circulation of CO_2 was from the buttom to the top of the extractor. The fatty material was then separated from the supercritical CO_2 by depression in separators and then recovered. Due to the solid nature of butter, temperatures of the extractor were maintained at 50 and 40°C for the sheanut kernel and *Canarium* pulp, respectively, while those of separators were 70°C for the shea butter and 60°C for the *Canarium* oil. The mass of the oil and CO_2 and the pressure of the

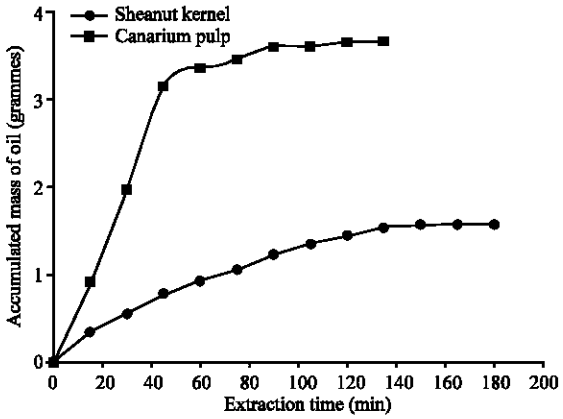

Fig. 1: Oil extraction curves of samples stored at -33°C after 1 month (sheanut kernel: Mc = 10.06 g; Qm = 749.67 g h^{-1} and *Canarium* pulp: Mc = 10.01 g; Qm = 837.33 g h^{-1})

separators were measured every 15 min during the extraction period. The extraction parameters are presented in Table 1. The end of the extraction is determined when the accumulated mass of the oil becomes constant (Fig. 1).

Hexane extraction: The hexane extraction was done during 9 h according to the soxhlet method (UICPA, 1979).

The extraction yield (% dry basis) was obtained from the following formula:

$$\frac{S*100}{MS}$$

Where S is the percentage by mass of the oil after extraction and MS the dry matter content of products used for the extraction.

Oil characterisation: The AFNOR (1981) norms were used to determine acid and iodine values.

The fatty acid composition was obtained after methylation of the oil, by gas chromatography using a Varian series 1400 device equipped with a manual injector, a Flame Ionization Detector (FID), a full column in melted silica of 1.5 m length, 0.125 mm diameter and 2 mm thickness and an integrator Spectra Physics SP 4100.

Statistical analysis: Tests were done in triplets. ANOVA was done on the results obtained using Statgraphics for windows Plus 3.0 software (1997). The difference between means was detected using the multiple comparison test of DUNCAN at a 95% confidence interval.

RESULTS AND DISCUSSION

Extraction yields: It can be observed from Table 2 that, whatever the extraction, solvent, the extraction yield for shea butter is significantly influenced by the duration and the storage temperature ($p = 0.05$). However, the hexane extraction yield for *Canarium* oil was only influenced by the period of storage and gave lower values at 18°C. The supercritical CO_2 extraction yields were lower than those obtained from hexane extraction, with a marked decrease for shea butter obtained in the first month.

The lower yields obtained with CO_2 can be explained by the lower water content of samples used for extraction. According to Royer (1999) yields of extraction of the sunflower and rape oils are as much higher as seeds are not dehydrated. A polarity effect can therefore be assumed: The polar water could facilitate the extraction of fat matter by non polar CO_2 through interactions with lipids. These interactions could be, by analogy with the extraction of caffeine (Wang, 1997) H_2CO_3-Huile type, the CO_2 being in the form H_2CO_3 in the supercritical state in presence of water. The solubility of water being nearly 100 times superior in the CO_2 than that in the hexane (0.01 kg m^{-3} in the hexane to 50°C and 1.35 kg m^{-3} in the CO_2 to 50°C and 125 bars). Another reason could be the lower solubility of triglycerides in the CO_2 compared to the hexane. Indeed, with the example of oleic acid (Chrastil, 1982; Bernard, 1995) it can be supposed that the solubility of unsaturated fatty acids is higher in the supercritical CO_2. The fat matter being essentially constituted of triglycerides, its solubility is decreased considerably because of the glycerol, in our experimental conditions. According to Francis (1954) the solubility of glycerol in liquid CO_2 is 0.5 g kg^{-1} (density of 650 kg m^{-3} and temperature of 298°K), however the liquid CO_2 is a "better solvent" than the supercritical CO_2 (Vallee and Barth, 1997). Garcia *et al.* (1994) found out that the heptane extraction yield of lipids, of by-products of cane sugar was superior to that obtained with the supercritical CO_2.

The loss of dry matter of the *Canarium* pulp, due to reactions of degradation encouraged by the higher dehydration of the pulp, could explain the progressive loss of the fat matter of fruits stored at 18°C.

Above 12 months, there is a strong deterioration of oil.

Studying the hexane extraction of sheanut butter from at ambient conditions (temperature of 24.5±1.5°C of and 94.5±2.5% of relative humidity) and that stored in a freezer (-18°C±2°C temperature and 55,5±8,5% relative humidity) for 2 months, Womeni (2004) reported, respectively, extraction yields of 33 to 41% and of 37-40%. These yields are probably lower than ours because of the higher storage temperature of this author which favours a decreased butter proportion. Indeed, he observed after 30 days of storage at ambient temperature, the development of moulds. With regard to *Canarium* fruits, after one week of storage at 22±1°C, Kapchie *et al.* (2004) obtained a hexane extraction yield of 37.50±0.85%. Although the storage period is only 1 week, this lower yield than ours after 1 month of storage, could be due to lower initial lipid content of fruits studied by these authors (38.00±0.96%).

Oil characterisation

Acid value: Acid values of the two lipids products extracted with supercritical CO_2 and hexane vary significantly ($p = 0.05$) with the storage time and conditions (Table 3). It observed that, with the 2 solvents, oil extraction yields of fruits stored at 18°C are higher than those of fruits stored at -33°C. This could be explained by the formation of hydroperoxydes under the influence of light and oxygen (autoxydation of polyunsaturated fatty acids by lipoxydases) at 18°C (Cheftel and Cheftel, 1984; MSDA, 2005; Weimer and Altes, 1989). The slight increase of the acid values of shea butter from nuts stored at -33°C could be due to the action of enzymes (lipases) during the defreezing period, encouraged by increase in the temperature and the fragile structure of the frozen products.

Table 2: Lipids yields of extraction with supercritical CO_2 and hexane (in%)

		Yield of extraction (%)			
		Shea butter		*Canarium* oil	
Storage conditions	Storage period (months)	CO_2	Hexane	CO_2	Hexane
T=18±2°C, Hr = 38±6%	1	28.11±0.50^{d2}	46.20±0.56^{d1}	26.22±0.23^{b2}	46.34±0.69^{a1}
	12	39.57±0.34^{a2}	53.15±0.41^{a1}	20.81±0.12^{c2}	35.84±0.44^{c1}
	24	34.84±0.40^{c2}	47.25±1.00^{c1}	16.10±0.41^{d2}	32.23±1.72^{d1}
T=-33±1°C, Hr = 37±6%	1	17.43±0.35^{e2}	44.88±1.40^{d1}	39.81±0.76^{a2}	43.68±0.14^{b1}
	12	38.47±0.41^{b2}	53.77±3.03^{a1}	40.45±0.44^{a2}	44.52±0.80^{b1}
	24	34.18±0.60^{c2}	49.91±1.22^{ab1}	25.66±0.36^{b2}	44.25±3.54^{b1}

T: Temperature; Hr: Relative humidity of the environment, means within columns with the same letter are not significantly different at p = 0.05, means within rows with the same figure are not significantly different at p = 0.05

Table 3: Acid value of lipids extracted with supercritical CO_2 and hexane

		Acid value			
	Storage	Shea butter		*Canarium* oil	
Storage conditions	period (months)	CO_2	Hexane	CO_2	Hexane
	1	56.77±5.22^{c1}	11.46±0.04^{c2}	102.63±0.98^{c1}	66.21±0.04^{c2}
T=18±2°C,	12	85.63±3.44^{b1}	16.46±1.14^{b2}	113.75±0.56^{b1}	113.89±0.84^{b1}
Hr= 38±6%	24	128.15±2.41^{a1}	18.80±0.73^{a2}	124.60±0.91^{a1}	120.81±2.98^{a2}
	1	19.88±0.56^{f1}	6.84±0.04^{e2}	19.01±0.09^{e1}	5.06±0.12^{f2}
T=-33±1°C,	12	21.22±0.38^{e1}	8.82±0.75^{d2}	19.44±0.29^{d1}	14.50±0.53^{e2}
Hr= 37±6%	24	23.07±0.39^{d1}	16.04±0.13^{b2}	20.17±0.83^{d1}	15.33±0.14^{d2}

T: Temperature; Hr: Relative humidity of the environment, means within columns with the same letter are not significantly different at p = 0.05, means within rows with the same figure are not significantly different at p = 0.05

Table 4: Iodine values of oils extracted with supercritical CO_2 and hexane

		Iodine value			
	Storage	Shea butter		*Canarium* oil	
Storage conditions	period (months)	CO_2	Hexane	CO_2	Hexane
	1	38.48±0.51^{a2}	75.19±0.34^{a1}	43.30±0.45^{b2}	96.98±0.52^{a1}
T=18±2°C,	12	27.74±0.65^{c2}	45.59±0.77^{c1}	34.42±0.55^{c2}	38.19±0.37^{d1}
Hr= 38±6%	24	21.68±0.68^{d2}	44.41±1.05^{c1}	27.12±0.11^{f1}	28.44±1.88^{e1}
	1	37.66±0.40^{a2}	62.59±1.83^{b1}	60.17±0.39^{a2}	95.09±0.79^{b1}
T=-33±1°C,	12	30.23±0.31^{b2}	43.40±0.40^{cd1}	33.58±0.14^{d2}	54.71±3.01^{c1}
Hr= 37±6%	24	22.57±0.54^{d2}	32.06±0.90^{e1}	29.10±0.90^{e1}	29.07±0.78^{e1}

T: Temperature; Hr: Relative humidity of the environment, means within columns with the same letter are not significantly different at p = 0.05, means within rows with the same figure are not significantly different at p = 0.05

The oils fatty materials extracted with supercritical CO_2 gave highest acid values, compared to the hexane extraction. This could be due to the higher solubility of free fatty acids in the CO_2. The substances with a lower content of OH groups are effectively more soluble in the non polar supercritical fluid (Peter, 1996). The quality of shea butters obtained with this solvent could be therefore, more dependent on the sheanut kernel quality.

The extraction with supercritical CO_2 gave butter with acid value superior to 0.6 (0.3% oleic acid), recommended for cosmetics (Kassamba, 1997).

To obtain oil with lower acid values, the storage of sheanuts and *canarium* fruits at -33°C is more appropriate.

Compared to the results obtained by Womeni (2004) at 24.5°C (27.85) after a 1 month storage period, ours are lower. This could be explained by the important load (packet of 2 kg) and, temperature and humidity (94.5%) conditions that would encourage a higher physiological activity of sheanuts. Kapchie *et al.* (2004) obtained out an acid index of *Canarium* oil after 1 week of storage at 22±1°C of 12.08±0.02. This value could be lower than the one reported here because of 1 storage month which could encourage a higher free fatty acid production.

Iodine value: Table 4 indicates the evolution of iodine value of lipids extracted with supercritical CO_2 and hexane as a function of storage period and temperature. These factors induced some significant differences between iodine values with 95% confidence. Generally, iodine values of oils extracted with supercritical CO_2 were lower than those extracted with hexane. The lower unsaturated degree of oils extracted with supercritical CO_2 could be due to the lower solubility of triglycerides in the supercritical CO_2.

After having also observed that the iodine value for shea butter were more higher during storage at 24,5°C than at -18°C, Womeni (2004) obtained values (of 100 and 94 at 24.5°C and of 85 and 73 at -18°C, respectively to the first and second months) which were superior to ours. This difference could come from fruits that had an initial value of iodine index of about 102, superior to ours of 77.59. As far as *Canarium* oils from pulp stored at 22±1°C after 1 week period, Kapchie *et al.* (2004) obtained a lower iodine index of 80.82±1.32. This difference from ours could be due to the lower initial index of the fresh fruit oil (86.52±1.07).

Fatty acids composition: Table 5 and 6 present the fatty acid compositions of shea butter and *Canarium* oils obtained with supercritical CO_2 and hexane for different storage periods and temperatures. Butter obtained with supercritical CO_2 contained exclusively palmitic, stearic, oleic and linoleic acids, with stearic acid (31.14-34.41% at 18°C and 34.29-36.74% at -33°C) and oleic acid (51.88 - 54.45% at 18°C and 50.67-53.66% at -33°C) as major constituents. The fatty acid profile of *Canarium* oils extracted with supercritical CO_2 indicated that they are mainly constituted of palmitic, oleic and linoleic acids. This proportion of palmitic acid at 18°C (52.96-58.29%)

Table 5: Fatty acid composition of shea butter extracted with supercritical CO_2 and hexane

	T = 18±2°C, Hr = 38±6%						T =-33±1°C, Hr= 37±6%					
	CO_2			Hexane			CO_2			Hexane		
Fatty acids (%)	1	12	24	1	12	24	1	12	24	1	12	24
C12				0.06						0.09		
C14				0.22						0.49		
C16	6.24	5.73	4.49	6.23	4.63	4.02	5.66	6.49	5.49	4.83	6.77	4.67
C16 : 1				0.14								
C18	31.14	34.41	33.67	31.06	26.87	36.43	34.39	36.74	34.29	31.32	25.60	33.02
C18 : 1	53.18	51.88	54.45	53.34	59.71	53.00	51.55	50.67	53.66	54.57	58.93	54.93
C18 : 2	9.44	7.98	7.39	8.69	8.41	6.26	8.40	6.11	6.56	8.46	8.37	7.13
C18 : 3				0.26	0.38	0.29				0.24	0.33	0.25
Total SFA	37.38	40.14	38.16	37.57	31.5	40.45	40.05	43.23	39.78	36.73	32.37	37.69
Total MFA	53.18	51.88	54.45	53.48	59.71	53.00	51.55	50.66	53.66	54.57	58.93	54.93
Total PFA	9.44	7.98	7.39	8.95	8.79	6.55	8.40	6.11	6.56	8.70	8.70	7.38

T: Temperature; Hr: Relative humidity of the environment; SFA: Saturated Fatty Acids; MFA: Monounsaturated Fatty Acids; PFA: Polyunsaturated Fatty Acids

Table 6: Fatty acid composition of *Canarium* oils extracted with supercritical CO_2 and hexane

	T = 18±2°C, Hr = 38±6%						T =-33±1°C, Hr= 37±6%					
	CO_2			Hexane			CO_2			Hexane		
Fatty acids (%)	1	12	24	1	12	24	1	12	24	1	12	24
C12				0.15						0.08		
C14				0.60	0.17	0.17				0.13	0.07	0.13
C16	52.96	58.29	55.60	40.95	48.42	53.67	37.25	33.37	35.99	37.60	35.90	34.93
C16 : 1				2.44	1.68		1.22	1.40		1.88	3.40	0.23
C18	2.28	3.45	3.15	1.71	2.84	3.84	1.80	1.63	1.84	2.11	1.75	1.98
C18 : 1	44.76	35.42	35.12	32.84	37.89	38.53	38.28	40.25	35.89	31.84	41.54	35.13
C18 : 2		2.84	6.13	20.77	8.57	3.67	21.45	23.35	26.28	25.57	16.86	26.39
C18 : 3				0.56	0.43	0.12				0.79	0.48	1.21
Total SFA	55.24	61.74	58.75	43.40	51.43	57.68	39.05	35.00	37.83	39.92	37.72	37.04
Total MFA	44.76	35.42	35.12	35.28	39.57	38.53	39.50	41.65	35.89	33.72	44.94	35.36
Total PFA		2.84	6.13	21.32	9.00	3.79	21.45	23.35	26.28	26.36	17.34	27.60

T: Temperature; Hr: Relative humidity of the environment; SFA: Saturated Fatty Acids; MFA: Monounsaturated Fatty Acids; PFA: Polyunsaturated Fatty Acids

was more than that at -33°C (33.27-37.25%). On the other hand, at -33°C there was higher proportion of linoleic acid (21.45-26.28%) compared to that at 18°C.

Compared to oils extracted with hexane, there are principal fatty acids in that extracted with CO_2, except linolenic acid. The absence of linolenic acid in the oil extracted with supercritical CO_2 let us suppose that they could rot less quickly than that extracted with hexane.

The stearic and oleic acids represent an important proportion of the total fatty acids of butters after 1, 12 and 24 months of sheanus storage at 18±2°C (84.4, 86.58 and 89.43%) and at -33±1°C (85.89, 84.53 and 87.95%). Compared to results on raw butter from Ngaoundere in Cameroon obtained by Kapseu *et al.* (2001) these 2 fatty acids had a closer proportion of 87.4% of the total fatty acids. This proportion is also closer to 86.85% obtained by Tano-Debrah and Ohta (1994) on the butter of shea kernels from Ghana which were stored for 6 weeks at 5°C. It is observed, in general, that these 2 fatty acids represent 84 to 89% of the total fatty acids of shea butter. Among the data presented in Table 6 on *Canarium* fruits, results obtained by Kapchie *et al.* (2004) after 1 week of storage at 22±1°C are closer to ours obtained from the hexane extraction after 1 month of storage at 18±2°C. Indeed, these researchers obtained proportions of palmitic, oleic and linoleic acids of 40.33, 31.81 and 20.95%, respectively. These three fatty acids represent 93.09% of the total fatty acids of *Canarium* oil for these authors, closer proportion to ours of 94.56%.

CONCLUSION

Compared to hexane, supercritical CO_2 gave under the experimental conditions studied lowest extraction yields, higher acid values and lower degree of oxidation. Shea butter obtained by supercritical CO_2 extraction was exclusively constituted of palmitic, stearic, oleic and linoleic acids, meanwhile *Canarium* oils extracted with supercritical CO_2 were constituted of palmitic, oleic and linoleic acids. The proportions of the principal fatty acids of *Canarium* oils extracted with CO_2 were closer to that of *Canarium* oils extracted with hexane at -33°C. These

results encourage greater investigations on supercritical CO_2 extraction of these fatty materials because of its selectivity.

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