

Fractionation Studies of Palm Oil by Density Gradient

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ABSTRACT

The paper describes a method of fractionating vegetable, animal and fish oils, and in particular palm oil. The method involves addition of a medium comprising two common solvents to the semisolid oils. On centrifugation, the olein and stearin are separated by the medium in the middle. Thirteen media made up from binary combinations of nine solvents, viz. water, propylene glycol, glycerine, methanol, ethanol, *n*-propanol, isopropanol (IPA), acetone and butanone, are found to be effective in olein-stearin separation. However, only the water/IPA and water/methanol systems have been studied in detail. The aqueous IPA provides a higher yield of olein than water/methanol but intersolubility between oil and medium is also greater. The fractionation process can be carried out at any suitable temperature. Fractionation of the special prime bleached (SPB) palm oil at 16 C yields an olein with a cloud point of 4.8 C. Some hybrid palm oils produce a large quantity of low cloud point olein which can be bleached readily. The process can be extended to include degumming and neutralization by using an alkaline medium for centrifugation. The olein fractions obtained have been found to be free of phosphatides and the free fatty acids reduced to as low as 0.02%. Metal-scavenging agents have also been added to the medium in an attempt to remove copper and iron. The development of this process into a continuous one has been demonstrated on the Alfa-Laval LAPX 202 Separator. Fractionation of crude palm oil using a density gradient provides seven fractions of different characteristics. The iodine values vary from 37.5 to 57.4 and the unsaturated fatty acids range from 32.7% to 51.2%. Triglyceride analysis by carbon numbers shows great differences in the C₄₈ and C₅₂ constituents of the fractions.

INTRODUCTION

The growth of the Malaysian palm oil industry in recent years has been remarkable. The overall oil palm hectareage has risen from 54,656 hectares in 1960 to 1,140,538 hectares in 1981 (1). Palm oil production showed a corresponding increase, rising to 2,824,464 tons in 1981 (2). Exports of both processed and unprocessed palm oil totalled 2,483,562 tons in that same year (2). The continued rapid development of the industry will result in an urgent need to find new markets or expand existing ones. This will entail greater product and quality research in order to meet consumer demands.

Fractional crystallization is one process which can be used to modify the characteristics of palm oil. It normally involves obtaining the right type of crystals by slow cooling of a palm oil melt or a solution of the melt and subsequent decantation, filtration or centrifugation of the slurry to separate the liquid oil from the solid. Three palm oil fractionation processes are used by refiners in Malaysia. They are dry fractionation, detergent fractionation and solvent fractionation (3).

Density gradient centrifugation is used extensively in the biochemical field for the isolation or purification of materials like viruses, subcellular organelles and whole cells, and is particularly useful in the sedimentation studies of macromolecules and synthetic polymers (e.g., refs. 4-6). The density gradient basically involves a column of fluid whose density increases towards the bottom. Separation is based on differences in sedimentation coefficient or in buoyant density of the materials in the mixture. The shapes of the gradient are varied and are important in the desired separation. Gradients can be linear in density and volume or otherwise. They can be continuous where the fluid density changes gradually and continuously along the gradient or they can be discontinuous (step gradients), in which case discrete layers having different densities can be discerned in the gradient. Gradients commonly used for biological separations are aqueous solutions of sucrose or of inorganic salts like cesium chloride. However such systems have been found unsuitable for fractionation of semisolid oils (7).

Our investigation into density gradient fractionation (8) was aimed at finding a process which can achieve efficient solid-liquid separation and yield palm fractions with possible new applications, while at the same time making minimal use of organic solvents.

EXPERIMENTAL

Materials

Crude palm and refined palm oils were commercial samples from Malaysia. Rice bran oil was from Malaysia Agricultural Oils and hybrid palm oils from United Plantations. Tallow,

TABLE I
Conditions for Effecting Olein-Stearin Separation by Centrifugation

Medium	Volume ratio of medium ^a					
	Crude palm oil	Refined palm oil	Rice bran oil ^b	Tallow	Lard	Herring oil
Water/acetone	35:65	36:64	34:66	35:65	38:62	35:65
Water/ethanol	43:57	43:57	42:58	46:54	44:56	41:59
Water/ <i>n</i> -propanol	44:56	44:56	44:56	47:53	46:54	46:54
Water/isopropanol	45:55	44:56	46:54	45:55	45:55	47:53
Propylene glycol/acetone	36:64	38:62	38:62	33:67	35:65	34.5:65.5
Propylene glycol/butanone	28:72	29:71	—	—	32:68	—
Propylene glycol/ethanol	47:53	47:53	46:54	48:52	48:52	44.5:55.5
Propylene glycol/ <i>n</i> -propanol	44:56	45:55	47:53	46:54	45:55	45:55
Propylene glycol/isopropanol	48:52	47:53	50:50	48:52	48:52	50:50
Glycerine/ethanol	24:76	25:75	23:77	25:75	25:75	21:79
Glycerine/ <i>n</i> -propanol	22:78	24:76	22:78	21:79	22:78	25:75
Glycerine/isopropanol	23:77	25:75	25:75	22:78	24:76	23:77
Centrifugation conditions (g/min)	2000/5	2500/5	2500/5	2500/15	2500/5	2000/5
Temperature (C)	25	25	25	25	25	3

^aThe volume ratio of oil to medium in each case was 1:1.

^bThe separation involved the oil and wax.

lard and herring oil were commercial products purchased from a local company (Georgetown Dispensary, Penang, Malaysia).

Olein-Stearin Separation

The general method involved addition of a suitable medium to the semisolid oil in the desired ratio and centrifugation of the mixture under predetermined conditions of temperature, *g* value of centrifugation and time required for separation (Table I). In some cases the oil had been recrystallized. In the usual procedure, palm oil was heated to 55 C followed by controlled cooling, with the temperature difference between oil and cooling bath maintained at 2 C down to the desired temperature.

The centrifuges used in the experiments were the IEC Model B20A, Haraeus Christ Cryofuge 6-6 and MSE LR-6.

Fractionation with Water/Isopropanol Containing Sodium Hydroxide

Water/IPA (40:60 by vol, 100 mL) containing 0.5 g sodium hydroxide was placed in a centrifuge tube. Crude palm oil (100 g) was added, the contents stirred thoroughly and then centrifuged. The olein was removed, washed with water (4 × 100 mL) and dried. The experiment was repeated with different amounts of hydroxide in the medium to determine the effective concentration for free fatty acids (FFA) removal.

Density Gradient Centrifugation

Density gradients were prepared in 17 cm × 25 mm id (85 mL) centrifuge tubes. The gradients were formed by layering fixed volumes of different water/IPA mixtures, with the highest density mixture at the bottom followed with progressively less dense mixtures above. For example, an effective gradient could be made by layering successively water/IPA mixtures (10 mL each) of compositions by volume of 44:56, 47:53, 50:50, 53:47, 56:44 and 59:41. To minimize turbulence of the layers, the mixtures were discharged down the side of the centrifuge tube through a funnel with a long stem drawn out to a fine tip. Crude palm oil that had been kept at ambient temperature of 26-30 C was carefully placed on the gradient and fractionated by centrifugation at 2000 rpm and 26 C for 30 min. The centrifuge used was a MSE LR-6 with a swinging bucket rotor.

Continuous Fractionation

Crude palm oil left at room temperature of 29 C and the same volume of water/IPA (45:55 by vol) were stirred thoroughly. The mixture was fed to the Alpha-Laval Laboratory Separator LAPX 202 equipped with a gravity disc of 53 mm diameter. The centrifugal speed was 8200 rpm. After 6-10 min, olein was collected from the middle nozzle of the separator and the medium from the upper one. When the flow of olein had stopped, the bowl of the separator was opened. Stearin and some medium were then purged out from the lowest nozzle.

Oil Characteristics

Cloud point was determined according to AOCS Method Cc 6-25, melting point Cc 1-25, cold test Cc 11-53 and peroxide value Cd 8-53 (9). Iodine values were tested based on the Wij's method with mercuric acetate catalyst (10). Free fatty acids, tocopherols, carotenes and anisidine values were analyzed using VOTC procedures (11). Totox value was calculated as 2 × peroxide value + anisidine value. Copper and iron were measured by atomic absorption spectrophotometry following the procedures of Siouffi (12) and Guillaumi (13). The Unilever method was used for bleachability

tests. Solid fat contents were measured on a Mk IIIA Newport Analyser with olive oil as reference (14). Triglyceride compositions according to carbon numbers were analyzed using a Hitachi Model 163 Gas Chromatograph. The column was 50 cm × 3 mm id glass packed with 3% OV-17 on 80/100 mesh Chrom W-HP. Programming was from 295 to 350 C at 3 C/min, with holding for 3 min at the initial temperature. The nitrogen gas flow was 90 mL/min.

RESULTS AND DISCUSSION

The first density gradient was prepared from glycerine and IPA. Crude palm oil was added, followed by centrifugation. It was observed that the olein appeared in one layer and the stearin in another. It was thought that, instead of centrifuging the palm oil with a density gradient, one could choose a suitable medium of the right density which is intermediate between the densities of the olein and stearin to achieve the same results.

Fractionation with an Oil-Insoluble Medium

Nine common solvents were selected for examination. Thirteen binary combinations of these solvents, viz. glycerine, propylene glycol, butanone, *n*-propanol, isopropanol, ethanol, methanol, acetone and water, were efficient in olein-stearin separation (Table I). Centrifugation of the crude oil with the appropriate medium effected separation into olein and stearin with the medium in between. This method of fractionation had been successfully tested on palm oil, palm kernel oil, rice bran oil, tallow, lard and herring oil. The optimum conditions for fractionation are summarized in Table I.

In a comparative study of the water/alcohol systems, it was discovered that the water/IPA combination provided the highest yield of olein (60%) from palm oil crystallized and fractionated at 20 C, followed by aqueous ethanol (49%) and aqueous methanol (41%). The introduction into the media of 0.5% of various surfactants and electrolytes prior to centrifugation made no significant improvement to the olein yield. The reagents used were Span 20, Span 80, Tween 20, Tween 40, Tween 80, sodium lauryl sulfate and magnesium sulfate.

When the water/IPA system was used for fractionation of palm oil, only ca. 0.3% of the oil was dissolved in the medium, whereas ca. 5% of the medium was present in the olein. This indicates essentially that the medium did not act as a solvent for the oil. Further experiments showed that the medium could be reused effectively up to seven times for separation.

Fractionation of Recrystallized Palm Oil

Although fractionation could be performed on unmelted palm oil at ambient conditions, further products were obtained by recrystallization of the palm oil at low temperatures, viz. 20, 18, 16 and 10 C, followed by centrifugation. The results are given in Table II.

The fractionation of some hybrid palm oils produced a large quantity of low cloud point olein (Table III) (15). Even though these oils possessed higher carotene contents than those of *E. guineensis*, bleachability tests showed that the oleins of such oils were readily bleached to below 2.0 red.

Removal of Free Fatty Acids, Gums and Metals

The addition of sodium hydroxide to the water/IPA medium prior to centrifugation enabled fractionation, neutralization and degumming to be achieved in one operation. In a typical case the presence of 1.0% sodium hydroxide in the medium reduced the free fatty acids in the olein from 1.9%

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TABLE II

Fractionation of Recrystallized Palm Oil at Low Temperatures by Centrifugation

Sample	Fractionation temperature (C)	Yield (%)	Olein		Stearin
			Melting point (C)	Cloud point (C)	Melting point (C)
Crude palm oil	20	70.0	21.5	6.8	52.5
Crude palm oil	18	60.0	20.0	6.0	52.0
Crude palm oil	16	48.0	20.0	5.5	51.0
SPB oil	20	72.0	21.0	6.5	52.0
SPB oil	18	60.0	20.0	5.0	50.0
SPB pil	16	45.0	20.0	4.8	47.0
Olein	10	17.3	19.5	4.0	26.5

Medium: water/IPA, 45:55 volume ratio. Palm oil/medium ratio (by vol), 2:1. Centrifugation condition: 14,900 g; 30 min.

TABLE III

Characteristics of Hybrid Palm Oils and Fractions

Hybrid	Fraction	Temperature (C)	Yield (%)	Melting point/cloud point ^a (C)	Carotenes (ppm)	Tocopherols (ppm)	Bleached oil color ^b (5/4 in. cell)
$F_1 \times E.g.^c$	Crude	20	—	28.5	398	473	—
	Olein		87.4	5.9	397	440	—
	Stearin		11.0	49.3	242	249	—
$F_1 \times E.g.^d$	Crude	15	—	30.0	362	492	—
	Olein		72.2	1.0	369	445	—
	Stearin		26.5	46.0	245	298	—
$F_1 \times F_1$	Crude	10	—	28.3	909	379	—
	Olein		72.9	0.7	948	333	—
	Stearin		24.9	41.3	724	303	—
<i>E.o.</i>	Crude	5	—	14.0	2366	718	4.5R 30Y
	Olein		86.2	4.2	2275	721	2.0R 30Y
	Stearin		12.6	36.0	1868	244	—
<i>E.o.</i> × <i>E.g.</i> (<i>Dura</i>)	Crude	19	—	28.0	1344	605	2.5R 30Y
	Olein		78.9	9.8	1261	451	1.5R 20Y
	Stearin		20.4	45.0	1087	252	—
<i>E.o.</i> × <i>E.g.</i> (<i>Pisifera</i>)	Crude	15	—	26.3	1156	532	1.8R 20Y
	Olein		87.7	1.7	1179	360	1.5R 20Y
	Stearin		11.8	49.0	671	239	—
<i>E.o.</i> × <i>E.g.</i> (<i>Dura</i>) × <i>E.g.</i> (<i>Pisifera</i>)	Crude	20	—	27.5	557	428	2.0R 20Y
	Olein		75.3	7.4	560	387	1.7R 20Y
	Stearin		21.7	47.5	383	—	—

^aMelting points for crude palm oils and stearins, cloud points for oleins.

^bUnilever method.

^c*E.g.*, *E. guineensis*.

^d*E.o.*, *E. oleifera*.

to 0.02% (Table IV). The phosphatide content of the olein was brought down to 0.00%. On fractionating crude palm oil with a higher FFA of 4.8%, an olein with 0.05% FFA was produced.

In view of the detrimental effect of trace metals—especially iron and copper—to the stability of palm oil, possibility of their removal using alkaline and acidic media for fractionation was investigated. The pH of the medium was adjusted by adding hydrochloric acid or sodium hydroxide. Table V shows that a strongly acidic medium was not very effective in removing such metals. However, as the medium was progressively made more alkaline, increasing amounts of copper and iron were found in the stearin. This was probably due to precipitation of metal hydroxides in the alkaline medium which were subsequently centrifuged down into the stearin.

In another experiment, metal-complexing agents were introduced into the water/IPA medium in an attempt to remove the copper and iron present in palm oil. The compounds used were acetylacetone, citric acid and biacetyl.

TABLE IV

Fractionation with Neutralization and Degumming

NaOH in medium (% w/w)	Sample	FFA (%)	Phosphatides (%)
—	Crude palm oil	1.82	0.19
0.0	Olein	1.94	0.00
0.5	Olein	0.04	0.00
0.6	Olein	0.04	0.00
0.7	Olein	0.04	0.00
0.8	Olein	0.02	0.00
0.9	Olein	0.02	0.00
1.0	Olein	0.02	0.00

There was no satisfactory removal of the trace metals.

Continuous Olein-Stearin Separation

The continuous separation of olein from stearin was dem-

onstrated on the Alfa-Laval Laboratory Separator, LAPX 202, with the water/IPA system. Employing a ratio of palm oil to medium of 1.6:1, olein was obtained through one exit, the medium through another and stearin with some solvents through the third. Thus three phases resulted under this condition. Data from experiments on fractionation of crude palm oil and recrystallized crude palm oil are presented in Tables VI and VII.

The continuous process was also extended to palm kernel oil (Table VIII) and hybrid oils. Fractionation of the F₁ hybrid palm oil produced an olein which might be suitable for use as salad oil and cooking oil (Table IX).

Density Gradient Centrifugation

When crude palm oil was centrifuged at ambient temperature with a density gradient of water/IPA, several products of varying properties could be obtained in one step. In one trial, a gradient of water/IPA mixtures of compositions 44:56, 47:53, 50:50, 53:47, 56:44 and 59:41 (volume ratios) separated palm oil into seven fractions (Table X). Carbon number analysis by gas liquid chromatography (GLC) indicated the significant changes in the C₄₈ (chiefly PPP) and C₅₂ (POO) constituents from the liquid to the more solid fractions (Fig. 1). However if the palm oil had been melted at 70 C and recrystallized by cooling gradually to 20 C, the same density gradient effected separation into

TABLE V

Copper and Iron Contents of Crude Palm Oil Fractions Obtained by Fractionation with Water/IPA Media of Different pH Values

pH of medium	Fraction	Copper (ppm)	Iron (ppm)
—	Palm oil ^a	1.15	19.3
2.8	Olein	0.67	14.7
	Medium	2.98	1.20
	Stearin	1.78	30.3
3.6	Olein	0.68	13.1
	Medium	2.94	1.04
	Stearin	1.78	39.9
5.2	Olein	0.67	13.2
	Medium	2.88	1.28
	Stearin	1.87	39.3
7.9	Olein	0.65	13.2
	Medium	2.85	1.04
	Stearin	1.82	41.5
10.5	Olein	0.55	9.88
	Medium	3.29	1.92
	Stearin	1.84	54.5
13.3	Olein	0.37	9.14
	Medium	5.16	5.76
	Stearin	3.23	58.0

^aCa. 1 ppm copper (as copper palmitate) and ca. 10 ppm iron (iron palmitate) had been added to increase the metals content of the starting palm oil.

TABLE VI

Fractionation of Crude Palm Oil by Alfa-Laval Separator LAPX 202^a

Oil: medium ratio	Sample	Olein yield (%)	Melting point/ cloud point ^b (C)	FFA	Tocopherol content (ppm)	Iodine value
No medium	Oil	—	—	2.32	646	54.1
	Olein	55.0	10.5	2.22	778	60.2
	Stearin	—	—	1.81	413	50.6
1:1	Oil	—	—	2.53	693	54.3
	Olein	72.5	11.8	2.83	790	59.2
	Stearin	—	53.0	2.76	495	45.0
1.6:1	Oil	—	—	2.01	857	55.3
	Olein	57.2	11.3	2.13	910	60.0
	Stearin	—	61.0	1.70	632	47.9
2:1	Oil	—	—	2.04	547	54.5
	Olein	73.1	11.8	2.28	710	57.1
	Stearin	—	54.5	1.58	384	43.9
3:1	Oil	—	—	2.50	568	54.8
	Olein	62.5	11.2	2.28	685	59.1
	Stearin	—	52.0	1.73	350	45.6
4:1	Oil	—	—	2.32	587	54.1
	Olein	66.3	10.5	2.27	731	59.6
	Stearin	—	52.5	1.77	352	46.2
5:1	Oil	—	—	2.32	670	54.1
	Olein	55.0	11.0	2.26	801	60.6
	Stearin	—	47.3	1.83	433	49.7

^aMedium: water/IPA, 45:55 volume ratio. Centrifugation: 8200 rpm at ca. 29 C.

^bMelting points for stearins, cloud points for oleins.

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TABLE VII

Fractionation of Recrystallized Crude Palm Oil by Alfa-Laval Separator LAPX 202^a

Oil: medium ratio	Sample	Olein yield (%)	Melting point/ cloud point ^b (C)	FFA	Tocopherol content (ppm)	Iodine value
No medium	Oil	—	—	2.01	635	53.2
	Olein	45.0	9.0	2.22	666	57.1
	Stearin	—	48.3	1.79	322	46.1
1:1	Oil	—	—	2.00	632	53.7
	Olein	56.3	11.3	2.20	682	57.2
	Stearin	—	52.3	1.65	350	46.3
1.6:1	Oil	—	—	2.27	560	53.4
	Olein	62.5	10.5	2.74	685	58.7
	Stearin	—	52.5	2.16	334	46.1
2:1	Oil	—	—	2.06	584	55.1
	Olein	65.6	11.4	2.23	710	57.0
	Stearin	—	51.5	1.86	358	45.1
3:1	Oil	—	—	2.10	600	53.5
	Olein	71.3	11.5	2.34	723	58.5
	Stearin	—	53.5	1.62	376	43.0
4:1	Oil	—	—	2.32	946	54.1
	Olein	68.8	9.5	2.20	999	60.0
	Stearin	—	53.0	1.74	630	47.2
5:1	Oil	—	—	1.98	663	53.7
	Olein	60.6	10.5	2.21	717	58.0
	Stearin	—	51.5	1.75	262	47.3

^aMedium: water/IPA, 45:55 volume ratio. Centrifugation: 8200 rpm at 20 C.^bMelting points for stearins, cloud points for oleins.

TABLE VIII

Fractionation of Recrystallized Crude Palm Kernel Oil Using the Alfa-Laval LAPX 202 Separator

Sample	Yield (%)	Dissolved medium(%)	FFA (%)	Melting point (C)	Cloud point (C)	Iodine value	Totox value
Crude oil	—	—	0.93	28.1	—	19.0	17.1
Olein	50.0	5.9	0.85	28.0	18.4	21.1	30.7
Stearin	50.0	5.6	0.72	29.9	—	17.9	25.55

TABLE IX

Fractionation of Recrystallized F₁ Hybrid Palm Oil^a Using the Alfa-Laval LAPX 202 Separator

Temperature of fractionation (C)	Fraction	Yield (%)	Cloud point (C)	Cold test (5½ hr) (C)	Iodine value	Carotenes (ppm)
—	Hybrid oil	—	—	—	64.0	1057
25	Olein	78.8	4.0	17	65.0	1073
	Stearin	—	—	—	54.2	808
20	Olein	81.1	2.5	15	65.8	1100
	Stearin	—	—	—	50.3	758
18	Olein	81.1	2.0	12	66.7	1115
	Stearin	—	—	—	47.8	679
16	Olein	80.0	1.5	10	65.8	1108
	Stearin	—	—	—	49.4	712

^a*Elaeis oleifera* × *E. guineensis*.

TABLE X

Characteristics of Palm Oil Fractions Separated by Density Gradient at 30 C

Fraction	Yield (%)	Carotenes (ppm)	Iodine value	Softening point ^a (C)	SFC ^b (%)
Olein: F1	61.8	662	57.4	19.4	3.0
Stearin: F2	6.2	615	52.9	33.4	10.5
F3	7.5	570	50.1	40.2	17.6
F4	3.5	526	46.5	47.0	25.8
F5	3.7	467	42.1	49.7	35.4
F6	4.9	434	40.0	51.5	40.6
F7	10.3	403	37.5	53.2	46.0
Palm oil	—	608	52.7	35.5	11.9

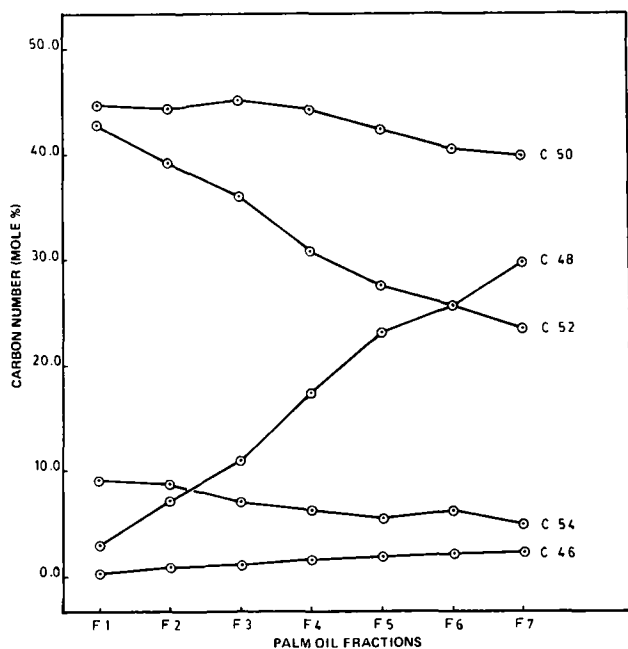
^aSoftening point, AOCS Method Cc 3-25.^bSolid fat content at 30 C by wideline NMR.

FIG. 1. Carbon number compositions by GLC of fractions separated by density gradient.

only one olein and one stearin fraction. It was believed that separation could have been influenced by different crystal forms and shapes of the palm oil.

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