Sharp-Melting Fat Fractions from Cottonseed Oil

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ABSTRACT

Solid fats of very short plastic range and melting points about equal to that of butterfat were obtained in yields up to ca. 64% by a single, fractional crystallization from hexane of byproduct stearines from the commercial winterization of cottonseed oil. One such product, consisting of approximately four-fifths 2-linoleodipalmitin and one-fifth 2-oleodipalmitin, melted almost entirely between 22 and 29 C, and was harder than butterfat at equal temperatures. Use as specialty fats is indicated.

INTRODUCTION

Approximately two-thirds of the cottonseed oil procured domestically is winterized to make salad oil. Winterization is generally accomplished by slowly cooling the refined oil to solidify an unwanted high-melting fraction and then removing this fraction by filtration. Fractional crystallization from a hexane solution (solvent winterization) is also practiced. The byproduct stearine or high-melting fraction, which amounts to ca. 10-25% of the oil processed and has a per pound value equal to about that of crude oil, is usually employed in the making of hydrogenated products.

This stearine would be expected to be rich in the disaturated triglyceride known to occur in cottonseed oil (1,2), particularly if the stearine is obtained by solvent winterization (3). Further fractionation of a stearine by partial crystallization from a solvent would be expected to yield a semisolid fat. Thus it should be possible to prepare relatively easily from cottonseed oil a semisolid fat without resorting to hydrogenation. Such a fat should have properties desired in a number of food uses.

EXPERIMENTAL PROCEDURES

Three stearines from the winterization of cottonseed oil were obtained from commercial processors. Each stearine was from a different processor and a different section of the country. Stearine A was the product of a solvent winterization, while stearines B and C were products of the more widely practiced nonsolvent winterizations. The iodine values and fatty acid compositions of the three stearines are recorded in Table I.

Further fractionation of each stearine to obtain a semisolid fat was accomplished by preparing a 20% solution by weight of stearine in *n*-hexane, cooling the solution to -15 C and holding it at this temperature for 12 hr, and then filtering to remove the liquid phase. The filtration, performed in a cold chamber at -15 C and with a stainless steel funnel equipped with a sintered, stainless steel element, was continued until the cake was dry. The cake was not washed.

In the subsequent discussion the products recovered from the cake and the filtrate will be identified by adding an *S* and an *L*, respectively, to the letter designating the type of stearine used. Thus fat A-S is the fat obtained as cake from the fractional crystallization of stearine A, and oil A-L is the corresponding oil recovered from the filtrate.

The butterfat and margarine oil used for comparison with the semisolid fats from the stearines were extracted from samples of USDA grade AA butter and a widely distributed brand of oleomargarine, respectively. The margarine oil had been manufactured from soybean oil. The procedure for separating the oil was the same in both cases. The butter (or oleomargarine) was heated to 60-70 C under vacuum and stripped with nitrogen to remove the moisture; the dried product was mixed with hexane, and the hexane solution was separated from the solids by filtration. The hexane was removed from the fat by heating under vacuum and stripping with nitrogen.

Well tempered samples of the several fats were examined dilatometrically (4), and the data obtained were used to calculate the proportions melted at various temperatures (5).

The hardness of well tempered samples of the fats was measured at different temperatures using a modification (6) of the Brinell method for metals. Relative hardness and

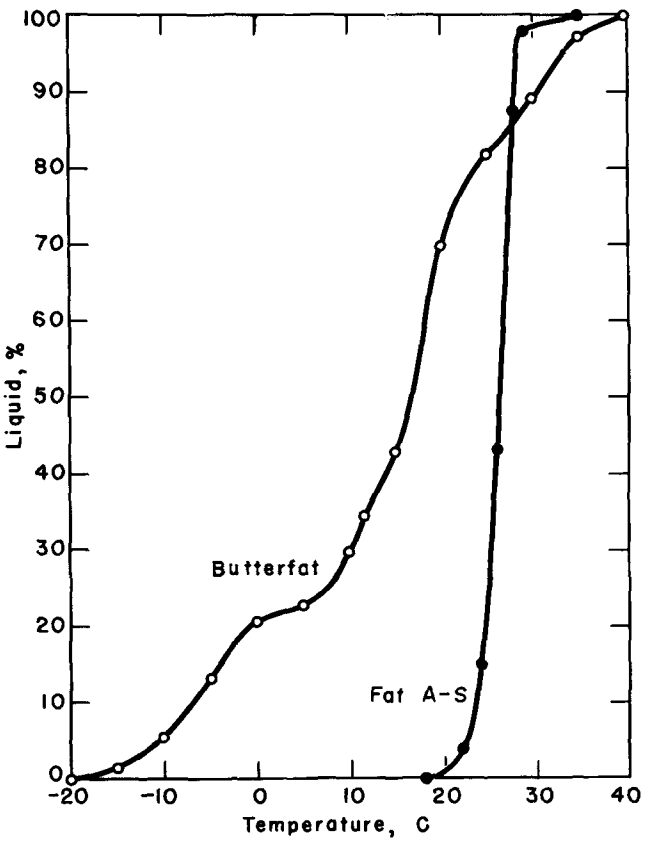

FIG. 1. Melting behavior of well tempered samples of butterfat and the solid fat fraction (A-S) from cottonseed oil stearine A.

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TABLE I

Analytical Data on Cottonseed Oil Stearines, Stearine Fractions and Butterfat

Product	Yield, %	Iodine value	Fatty acid composition[a], wt %				
			Myristic	Palmitic	Stearic	Oleic	Linoleic
Stearine A							
Original	—	71.5	0.7	50.3	2.0	10.6	36.5
Fat A-S	64.5	55.7	0.2	64.7	0.7	7.1	27.3
Oil A-L	34.5	103.4	1.2	25.8	2.8	15.8	54.4
Stearine B							
Original	—	97.8[b]	0.5	33.3	2.5	13.8	49.5
Fat B-S	39.6	58.1[b]	0.4	60.3	1.8	7.5	29.8
Oil B-L	59.9	120.4[b]	0.7	18.2	2.7	17.2	60.6
Stearine C							
Original	—	104.2	0.6	29.8	2.1	13.9	53.1
Fat C-S	31.3	60.9[b]	0.5	57.5	2.0	7.8	31.9
Oil B-L	68.7	122.5[b]	0.7	17.7	2.2	16.4	62.3
Butterfat	—	29.7[b]	11.2	28.9	13.9	28.9	1.5

[a]Only major fatty acids are reported. Butterfat contained at least 17 additional fatty acids.
[b]Calculated from fatty acid composition.

brittleness were measured in a less precise manner, as described below.

The fatty acid composition of each of the products was determined by well established gas liquid chromatographic procedures.

RESULTS AND DISCUSSION

Cottonseed oil contains virtually no trisaturated glycerides (1). Mattson and Lutton (7) found ca. 90% of the fatty acids in the 2 position of cottonseed oil triglycerides to be unsaturated. In previous work with a stearine almost identical to stearine A and obtained from the same processor, we found over 90% of the fatty acids in the 2 position to be unsaturated and the stearine to contain less than 0.3% trisaturated glyceride (8). These facts, combined with the finding that the fatty acids from fat A-S contained 64.7% palmitin acid, 27.3% linoleic acid and 7.1 oleic acid (Table I), indicates that the fractionation of stearine A produced a solid fat product (fat A-S) consisting of about four-fifths 2-linoleodipalmitin and one-fifth 2-oleodipalmitin. This solid fat, which had an iodine value of 55.7, was obtained in the relatively high yield of 64.5%.

Fractionation of stearines B and C produced solid fats B-S and C-S, respectively, which had a fatty acid composition similar to that of solid fat A-S (Table I). However the yields were considerably lower. This was to be expected, because the winterization of cottonseed oil without the aid of a solvent resulted in the occlusion of some low-melting oil with the fat crystals. The solid fat from stearine A was in effect derived from cottonseed oil by two crystallizations from a hexane solution, while the solid fats from stearines B and C were derived from cottonseed oil by one crystallization without solvent and one crystallization from a hexane solution.

The low-melting oil fractions (oils A-L, B-L and C-L, Table I) recovered from the filtrates were, of course, similar to unfractionated cottonseed oil in fatty acid composition. Presumably these fractions could be used as cottonseed oil.

The percentages of the fats liquid at given temperatures, calculated from dilatometric data on well tempered samples, are recorded in Table II. Percentages for well tempered samples of the butterfat and margarine oil are included for comparison. All three solid fats from the stearines exhibited a very short melting range, with the shortest from stearine A. The butterfat exhibited a considerably longer melting range, while the margarine oil exhibited a very long melting range. The remarkably short melting range of fat A-S is compared graphically with that of the butterfat in Figure 1.

Hardness-temperature relationships for the solid fats from the stearines and those for the butterfat and margarine oil are recorded in Table III. All samples were

TABLE II

Percentages of Liquid in Fats at Various Temperatures[a]

Temperature, C	Liquid content, %				
	Fat A-S	Fat B-S	Fat C-S	Butterfat	Margarine oil
-20	—	—	—	0.0	2.1
-15	—	—	—	1.6	12.9
-10	—	—	—	5.5	23.2
- 5	—	—	—	13.4	30.3
0	—	—	—	20.7	32.3
5	—	—	0.0	22.7	36.6
10	—	0.0	1.2	29.8	43.1
15	—	1.4	3.0	43.0	51.9
18	0.0	4.1	6.0	58.1	56.6
21	2.1	10.9	15.1	73.0	62.3
23	7.3	23.1	28.9	79.6	63.9
26	43.2	74.2	80.8	83.3	68.5
29	98.0	92.6	94.6	87.8	74.3
31	99.0	94.0	95.8	90.7	78.9
33	99.5	95.8	97.2	94.3	84.5
36	100.0	98.1	99.4	98.6	91.9
39	—	100.0	100.0	100.0	98.2
41	—	—	—	—	100.0

[a]Well tempered samples.

TABLE III

Hardness of Fat Products[a]

Temperature, C	Hardness index[b]				
	Fat A-S	Fat B-S	Fat C-S	Butterfat	Margarine oil
7	17.5	6.8	5.9	8.6	1.4
10	15.8	5.5	4.5	5.5	1.0
12	11.4	5.2	3.7	4.0	0.9
14	9.5	5.2	3.0	2.8	0.6
16	7.8	3.2	1.9	---	---
18	5.1	3.0	1.5	---	---
20	4.0	1.9	0.5	---	---
23	2.4	1.1	---	---	---

[a]Well tempered samples.
[b]Measured as kg/cm^2 (Reference 6).

tempered well before being tested. Two of the fats, B-S and C-S, had a hardness-temperature relationship quite similar to that of butterfat. The other fat, A-S, was much harder than the butterfat at all of the test temperatures. The margarine oil was quite soft at all temperatures, which would be expected on the basis of its liquid content at these temperatures.

When samples of the fats from the stearines were stored at room temperature and then placed in the mouth, each melted with a pleasing, cooling sensation.

Because coconut oil is frequently incorporated in formulations for enrobing frozen confections, semiquantitative measurements of brittleness were made at -22 C with samples of edible coconut oil and the solid fats from the stearines. Freshly cleaned steel strips (0.14 mm thick by 20 mm wide by 152 mm long) were dipped into samples held at 36 C and then quickly solidified at -22 C. After ca. 1 hr the samples were examined for brittleness by slowly reflexing each strip. The coconut oil shattered immediately; fat A-S shattered after moderate flexing; fat B-S cracked slightly after considerable flexing, and fat C-S did not crack after considerable flexing.

In related tests, a needle was pushed into small discs of the fat samples solidified rapidly at -22 C and held at this temperature. The coconut oil was the hardest and most brittle. Fat A-S appeared to be nearly as hard but not as brittle. Both fats B-S and C-S were considerably softer and more waxy, with fat C-S the softer and least waxy of the two by a small margin.

One can conclude, on the basis of the data obtained, that normally solid fats rich in 2-linoleodipalmitin and 2-oleodipalmitin can be produced easily from the stearine obtained as a byproduct in the winterization of cottonseed oils. These fats have melting points about equal to that of butterfat but possess a much shorter plastic range. They should have utility in the formulation of special food products.

REFERENCES

1. Hilditch, T.P., and L. Maddison, J. Soc. Chem. Ind. 59:162T (1940).
2. Riemenschneider, R.W., C.E. Swift and C.E. Sando, Oil and Soap 17:145 (1940).
3. Cavanagh, G.C., E.J. Cecil and K. Robe, Food Proc. 22 (4):38 (1961).
4. Lovegren, N.V., and R.O. Feuge, JAOCS 42:308 (1965).
5. Singleton, W.S., and A.E. Bailey, Oil Soap 22:295 (1945).
6. Lovegren, N.V., W.A. Guice and R.O. Feuge, JAOCS 35:327 (1958).
7. Mattson, F.H., and E.S. Lutton, J. Biol. Chem. 233 (4):868 (1958).
8. Feuge, R.O., B.B. Gajee and N.V. Lovegren, JAOCS 50:50 (1973).

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