Development of a Continuous Process to Obtain a Confectionery Fat from Tallow: Final Status

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ABSTRACT

A two-step pilot plant continuous process fractionally crystallized edible beef tallow into three fractions—a confectionery fat (14%), cooking oil (69%), and stearine (17%). The tallow was dissolved in acetone to a concentration of 11.6% by weight. The use of scraped surface heat exchangers with high external recycle permitted crystallizations at 60-65 F and 40-42 F, respectively, with only 5 min nominal dwell times in each crystallizer. A cost estimate indicated that the process to separate continuously a confectionery fat from tallow may be commercially feasible, dependent upon the price and market of the other two fractions.

NOMENCLATURE

C = wt fraction of tallow in stream at any time; Co = wt fraction of tallow in stream before washing; F = volume of feed; FFA = free fatty acid, %; IV = iodine value; PPY = pounds per year; ST = stearine fraction; TCB = tallow cocoa butter or confectionery fat fraction; W = volume of wash; τ = residence time.

INTRODUCTION

Cocoa butter is the highest quality confectionery fat and the standard of the chocolate industry. In recent years, the price of imported cocoa butter has risen sharply, and, with the advent of the Chinese market (1), the prospect of cheaper cocoa butter is slim.

Tallow is usually the lowest-priced fat or oil in the world (2). Luddy et al. (3) fractionated edible beef tallow "by an acetone crystallization procedure to yield 5 fractions: 2 solid glyceride fractions comprising 14% (ca. 7% each) of the original tallow; 2 liquid fractions of 59% and 7%; and one semisolid fraction of 20%. The semisolid glyceride fraction was similar to cocoa butter." From this stage, we began a study to develop a commercially feasible continuous process to separate edible tallow into 3 fractions: a solid fraction, a liquid fraction and a semisolid fraction.

EXPERIMENTAL PROCEDURES

Analytical

Prior to submission for analysis, samples of the stearine fraction (ST) and the confectionery fat fraction (TCB) were stripped of acetone in a tray dryer at 180-190 F for several hours. The bulk of the solvent was removed from the oil fraction by evaporation in a Precision Glass Evaporator prior to either deodorization or stripping in the tray dryer. Stream concentrations were determined by sample weights before and after acetone removal.

Iodine value (IV) of each fraction was determined by the AOAC (Hanus) method (4). Free fatty acid (FFA) content of the oil fraction was determined by AOCS method Ca-5a-40 (5). Peroxide value of the oil fraction was measured by AOAC method 28.022 (6).

Water content of the reagent grade acetone was determined by the method of MacDonald and Brady (7) as modified by Egoville and DellaMonica (8).

The thermal characteristics of the ST and TCB fractions were measured on a Perkin Elmer Model DSC-2 Differential Scanning Calorimeter equipped with a Perkin Elmer Model 56 single-pen recorder. Operating parameters were: heating rate -10 K/min, range -10 mcal/sec, and sample size, ca. 10 mg. All TCB samples were tempered for at least 48 hr at 20 C. The ST fractions were tested untempered.

Materials

Rustco Products Co., Denver, CO, supplied filtered, deodorized edible tallow containing BHA, BHT and citric acid. The tallow had an FFA content of 0.25%, peroxide value of 1.4 and a fatty acid distribution as listed in Table I. We purchased certified ACS grade acetone, ΔA-18, from Fisher Scientific Co., Somerville, NJ. Recovered and recycled acetone was monitored and found to contain 0.6 to 1.0% water and no nonvolatile residue.

Equipment and Procedures

We are reporting on 2 process lines: a semicontinuous line with a batch filter and a continuous line with a continuous filter. We studied the process with only half the major equipment by running the 2 crystallization/filtration steps on separate days.

The process and surrounding research area conformed to class I, group D, explosion-proof construction. Seals, packings, gaskets and o-rings were Teflon because Teflon is acceptable for food use and impervious to acetone. The process line was ½ in. ss tubing or pipe.

Semicontinuous Process

Figure 1 is a schematic of the semicontinuous process. We used a Lapp Pulsafeeder diaphragm pump to feed the acetone and a Zenith gear pump to feed the tallow. The 2 streams were mixed at a temperature that ensured a homogeneous solution containing no crystals.

The crystallizer consisted of two 0.7-ft^2 scraped surface heat exchangers (Votators), in series, with a centrifugal pump (Eastern 3J-34-G) to supply high recycle. (We needed 2 heat exchangers to obtain sufficient volumetric hold-up and heat transfer surface to match the feed rate.) The heat exchangers had Teflon scraper blades. The rotor speeds in

TABLE I

Fatty Acid Distribution of Tallow

Fatty acid	%
14:0[a]	3.4
16:0	27.4
16:1	3.7
18:0	23.0
18:1	40.7
18:2	Trace

[a]Number of carbons:number of double bonds.

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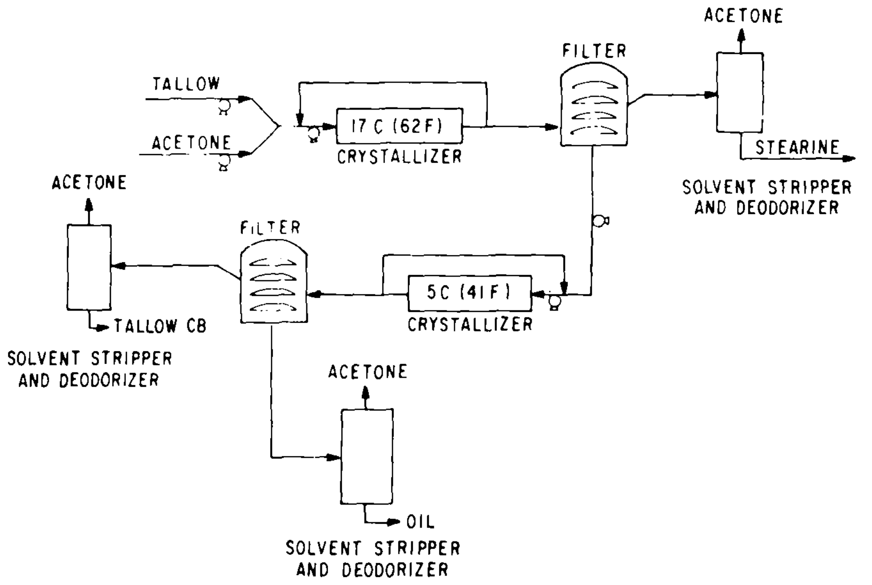

FIG. 1. Semicontinuous process flow sheet.

the 2 scraped surface heat exchangers were fixed at 100 and 160 rpm, respectively. A pneumatic control valve maintained low positive pressure in the crystallizer to prevent localized expansion flashing and crystallization. A Bell & Howell transducer measured the pressure. A pneumatic control valve (Foxboro) throttled the coolant to maintain the process temperature set point.

Fitted with type A-3 paper, a jacketed Sparkler filter (14S11) separated the slurry into a filter cake containing stearine and a filtrate containing TCB and oil. The filtrate was stored in a 55-gal ss kettle until further processed.

We used an Eco lobe pump to feed the filtrate to the second crystallizer (the same equipment as the first). This pump failed to maintain a steady flow rate and was replaced in the continuous process. Except for the temperature, the second crystallization and filtration were duplicates of the first. When the feed was exhausted, wash acetone, pumped through the crystallizer to the filter to maintain the same temperature for wash as for crystallization, flushed the residual mother liquor from the filter cake.

Continuous Process

A 1-ft^2 vacuum drum filter (Bird-Young YUF-101) supplanted the batch filter in the continuous process (Fig. 2). We added a wash pan and a Teflon doctor blade. Because this filter was not originally designed to filter a volatile fluid such as acetone, we enclosed it. Flashing and consequent cloth binding are problems unless there is a saturated solvent atmosphere in the filter enclosure. We installed a Nash sealed ring vacuum pump to recycle acetone exhaust vapors to the enclosure to create a solvent atmosphere. Unfortunately, the enclosure was not sufficiently vapor-tight to achieve a positive pressure and a saturated solvent atmosphere.

To feed the filtrate from the first crystallization to the second crystallizer, we replaced the lobe pump with a Lapp Pulsafeeder diaphragm pump. The rest of the continuous

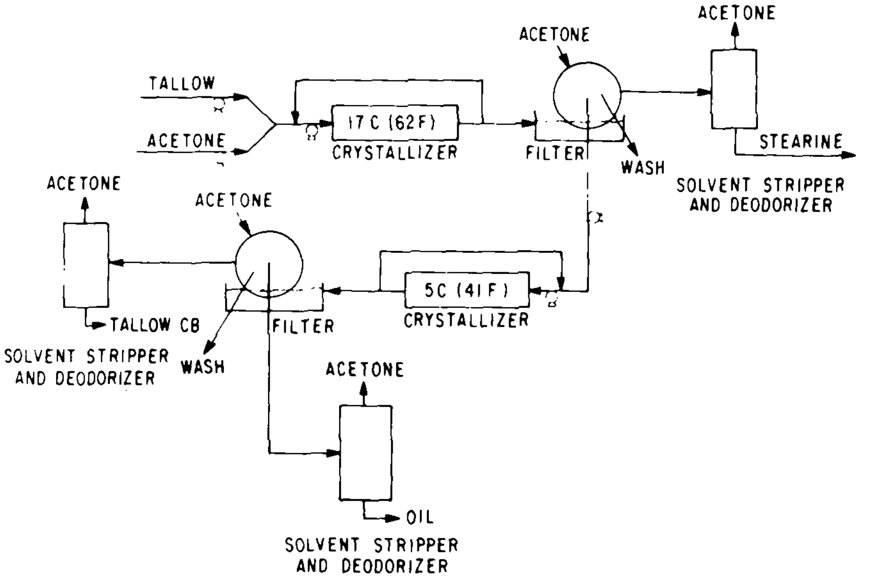

FIG. 2. Continuous process flow sheet.

process line was the same as the semicontinuous line.

RESULTS AND DISCUSSION

Batch crystallization, with dilute solutions and long dwell times, requires large solvent inventories. The solvent/tallow ratio should be high to achieve good selectivity in the fractionation. Luddy et al. (3) used a volume ratio of 10:1, acetone/tallow. Their work was the starting point for our research; we began at approximately the same concentration, 9.1% by wt. We increased the tallow concentration somewhat during this study to 11.6% by wt.

The laboratory batch fractionation procedure consisted of 4 crystallizations giving 5 fractions which, when combined, yielded 3 products. Four crystallizations require 4 similar process lines within the overall process. We tried reducing the number of pieces of the capital equipment in half by reducing the number of crystallizations from 4 to 2. It worked. Instead of 4 crystallizations and 5 fractions yielding 3 products, 2 crystallizations give 3 fractions and 3 products.

Cooling, crystallizing, and equilibrating each require a long time on a commercial, batch-scale fractionation. For instance, that which took 30 min to cool and 4 hr to equilibrate may require 4 hr and 16 hr, respectively, on the commercial scale. Even then, the production may be different because of different thermal history and hydraulic pressure. There is batch-to-batch variation to contend with. Solvent history must be very large because of the long residence time. Crystallizing/holding vessels must be large and numerous.

What if the crystallization were continuous and of short residence time? Equipment and solvent inventory would be smaller. The process would be more reproducible and more easily scaled-up. If the residence time distribution were reproduced, scale-up would be direct.

To obtain a good confectionery fat, the feed must be saturated with seed crystals. We recycled over 95% of the slurry to provide seed crystals. Such a high recycle produced a high ratio of back-mixing to plug flow and eliminated any detectable temperature rise from the entering feed stream. When we reduced the recycle to increase plug flow, product quality deteriorated.

Figure 3, curve A, is an experimentally determined solubility curve for tallow. We found this curve worthless for predicting the temperature/concentration relationship for this system during crystallization. By trial and error, we found that 62 F (17 C) and 41 F (5 C) gave fractions corresponding to a stearine and a confectionery fat, respectively. Within limits of experimental error, these temperatures are valid over a broad feed concentration range. But what of the solubility curve? We found that crystallization followed, for want of a better term, operating lines, B. There is a family of operating lines, B, each established by the initial solution concentration. These lines can be used to predict the temperature/concentration relationship during crystallization.

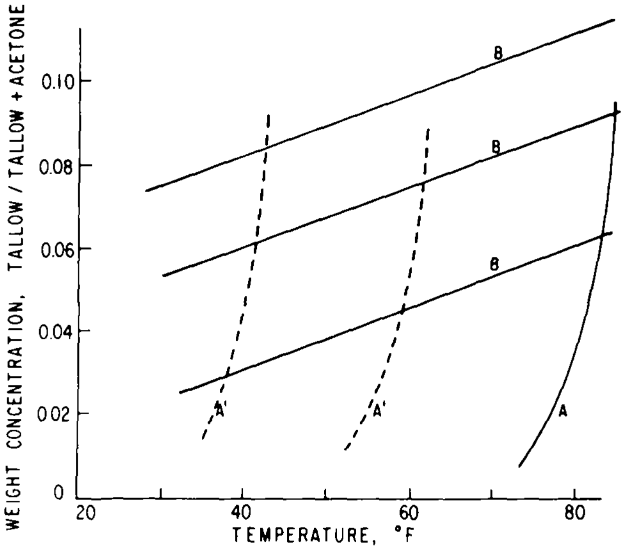

FIG. 3. Solubility of tallow in acetone.

Why? As is well known, tallow is not a pure compound but a complex mixture of triglycerides. The tallow solubility curve, A, establishes only the onset of crystallization for the least soluble compound, e.g., tristearine, under the influence of the mutual solubility of the other triglycerides. During processing, tristearine and other less soluble triglycerides are removed as the ST fraction. The remaining fat is no longer tallow but a fractionated tallow with a different solubility curve, e.g., A'. In fact, a different solubility curve corresponds to each fractionated tallow. Fortuitously, these curves, A', are roughly parallel to the tallow solubility curve, A. It is the intersection of an operating line, B, as established by the original solution concentration, and a solubility curve, A', which determines the filtrate concentration at a specific temperature. The more soluble triglycerides are in solution in the filtrate and the less soluble are predominantly crystallized. The triglyceride fraction

TABLE II

Semicontinuous Process Parameters and Results

Run #	Feed flow rate g/min	Concentration (g tallow) wt tallow / wt tallow + wt acetone	Crystallization temperature F	(C)	Residence time min	Yield % ST	% TCB
1A[a]	349.3	0.096	65	(18)	4.8	17.7	
B	160-556	0.078	41	(5)	3-10.5		14.8
2A	331.5	0.103	65	(18)	5.1	17.2	
B	270-449	0.083	42	(6)	3.7-6.3		14.9
3A	335	0.092	63	(17)	5	18.5	
B	335-280	0.072	42	(6)	5-6		11.3
4A	335	0.102	62	(17)	5	17.6	
B	280	0.082	41	(5)	6		13.7
5A	335	0.096	62	(17)	5	14.6	
B	419	0.082	41	(5)	4		11.5
6A	365-729	0.116	60	(16)	2-5	16.4	
B	210	0.097	40	(4)	8		16.4

[a] A—first day's run; B—second day's run.

crystallizing at and above 62 F (17 C) forms the ST fraction. The fraction crystallizing above 41 F (5 C) and below 62 F (17 C) constitutes the TCB fraction. The fractionated tallow which remains in solution at 41 F (5 C) makes up the oil fraction.

This solubility behavior is both an advantage and a disadvantage. Temperature control is simplified because a few degrees variation is insignificant to product quality. A slightly variable feed concentration will not upset the process or alter the quality of the fractions. However, changing the concentration will not permit a more favorable, energy-saving crystallization temperature.

Our preliminary work on a continuous process gave encouraging results at just 30 min dwell time per crystallizer. Eventually, we found nominal dwell times of 5 min acceptable. It might even be possible to reduce these times a little more with additional research.

Table II lists process parameters and results for 6 of the runs in the semicontinuous process. The crystallization temperature range for stearine was 60-65 F (16-18 C) and 40-42 F (4-6 C) for the TCB fraction. The nominal residence time in the first crystallizer (stearine) was 5 min. The nominal residence time for the second crystallization (TCB) varied from 3 to 11 min because the lobe pump failed to maintain a steady flow rate. The recycle pump ran unthrottled, recycling over 97% of the process stream. Figure 4 presents the results of a residence time distribution study for this system (9). Integrating the area under the curve from $\theta = 0$ to $\theta = 2$ and normalizing, we found backmixing accounts for 74% and plug flow, 26% of the flow.

Yields averaged 17% for ST and 14% for TCB. These yields were calculated using the inlet and outlet stream concentrations. Yields determined from the actual weight of filter cake recovered would be greater by the amount of mother liquor absorbed on the cake. Figure 5 presents a typical DSC curve for the TCB fraction. The melting profile resembles that of cocoa butter. The melting peak is slightly higher than that of cocoa butter, and there is a minor lower melting component. This lower melting component is undoubtedly oil absorbed by the filter cake and not removed in the washing step. The IV of the TCB fraction averaged 34 (values of 30 to 39). Luddy et al. (3) showed that the confectionery fat fraction separated from tallow had a fatty acid composition similar to cocoa butter with about 1/3 unsaturated acids. They also showed that it was compatible with cocoa butter. DSC scans of 1:4 blends of TCB from the semicontinuous process with cocoa butter showed essentially no change in the DSC scan from cocoa butter, indicating that TCB is compatible. The IV of the oil was 58 with a peroxide value of 0. The FFA content was less than 0.5% before deodorization.

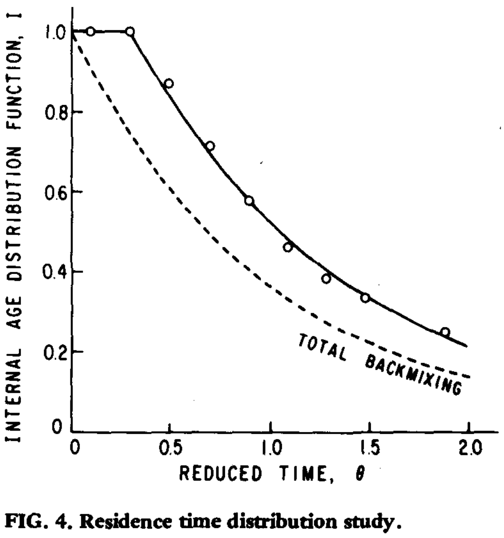

FIG. 4. Residence time distribution study.

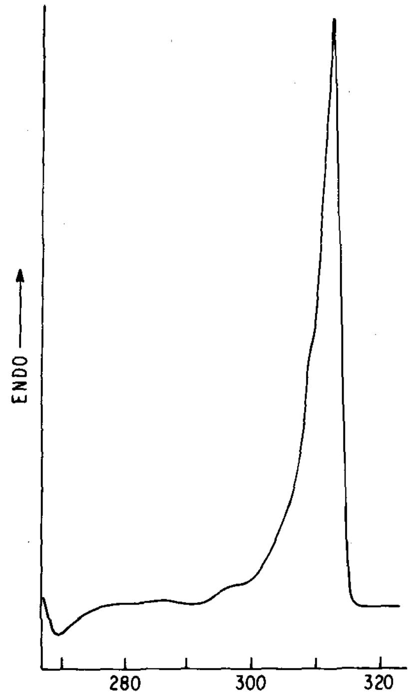

FIG. 5. DSC of TCB from the semicontinuous process.

What would be the selling price of the TCB, the oil and the stearine for a grass-roots plant? We made a cost estimate based on this semicontinuous process using 24 hr/day, 350 day/year processing, and 20% net annual return on fixed capital. The selling price of any one product depends on the selling price of the other 2. Commercial feasibility depends on the market prices and potential for the oil and stearine fraction. Stearine and TCB are by-products. The stearine selling price was assumed equal to the cost of edible tallow. Oil was assigned a premium of 6¢/lb. The cost of edible tallow was 21¢/lb at the time of the cost estimate. Tallow for 21¢/lb, oil at 27¢/lb, and stearine for 21¢/lb—these prices were the basis for the cost estimate.

Figure 6 shows the TCB selling price for this grass-roots plant as a function of tallow cost and production level. With tallow at 21¢/lb and production at 20 × 10^6 lb/yr, the TCB selling price would be $3.44/lb. Figure 7 shows the

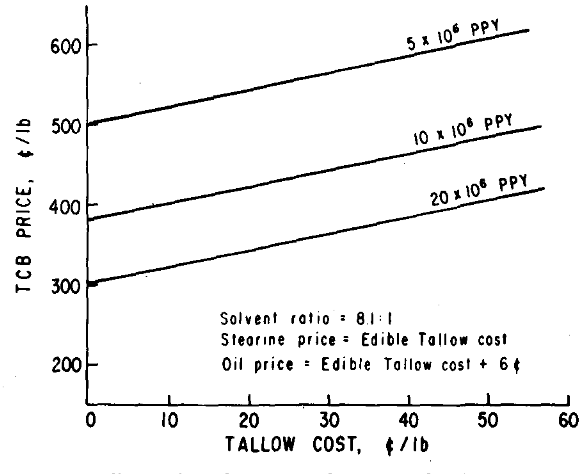

FIG. 6. Selling price of TCB as a function of tallow cost.

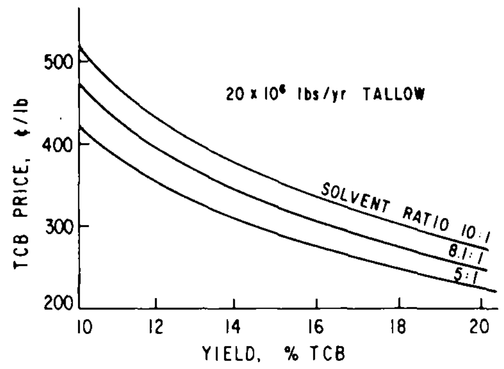

FIG. 7. TCB selling price vs TCB yield.

effect of TCB yield and solvent ratio.

We had little difficulty filtering with the batch filter. Pressure drop was less than 5 psi with a 2" filter cake. Filtrates were clear. However, the TCB fraction contained some low melting material—the oil fraction. Washing all the residual mother liquor out of the thick porous filter cake is difficult. On the other hand, a vacuum drum filter operates in a continuous manner and provides a thin cake for washing. We therefore tried a continuous process with a vacuum drum filter.

A thin, uniform, somewhat porous filter cake, a good solvent spray system and the proper wash solvent rate are necessities for an efficient wash system. To determine the required amount of washing, we monitored filtrate concentration from the Sparkler filter during washing in the semicontinuous process. We combined and correlated the data from 8 runs. Figure 8 shows that the filtrate concentration decreases exponentially ($C/C_0 = e^{-2.54(W/F)}$) and is fairly steady after W/F = 0.5. We therefore used a wash solvent rate equal to half the feed rate.

The drum filter, as modified and enclosed, was not sufficiently vapor-tight to create a saturated solvent atmosphere. The filtrate flashed and caused the cloth to blind.

Filter capacity far exceeded the crystallizer capacity. To obtain a continuous filter cake, 3/16 in. (4.8 mm), we ran very slow drum speeds—0.1-0.5 rpm. The long dwell time in the filter trough permitted settling, so we added a rake to the trough.

The high recycle rate and agitation from the recycle pump generated small, difficultly filterable crystals. No cloth tried was sufficiently retentive. The 2 best, Amatek 100% polyester cloth XBH-20M2-HD8 and Tetko Inc., Pecap Screening Fabric, Swiss Polyester Monofilament HD7-1, were marginally effective. Slight changes in the crystal size distribution destroyed the filtration. Decreasing recycle increased crystal size and produced better filtration, but product quality deteriorated.

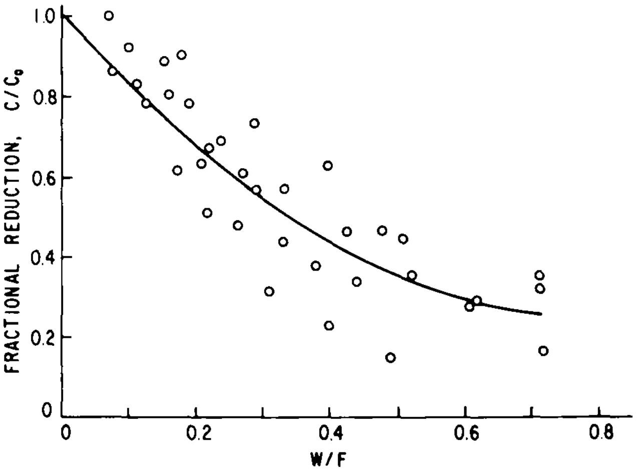

FIG. 8. Filtrate concentration as a function of the wash/feed ratio.

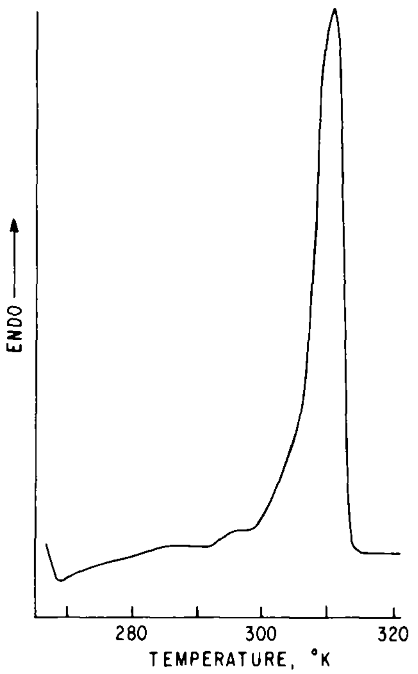

FIG. 9. DSC of TCB from the continuous process.

Some continuous process runs gave TCB fractions which were promising, e.g., DSC in Figure 9. Although the bulk of the fraction melted in the proper range, some low melting material was present. Yields were low at 9% TCB and 20% ST. We used the amount of filter cake collected in a measured period to calculate yields. Crystallization temperatures were 61 F (16 C) and 41 F (5 C). The crystallizer nominal residence times were 3.5 min for ST and 6 min for TCB. The feed concentration was 11% tallow by weight.

Our projected TCB selling price of $3.44/lb is limited to commercial-sized duplicate of the semicontinuous process. A commercial process would preferably be continuous.

ACKNOWLEDGMENTS

R.L. Stabile made the cost estimate. E.S. DellaMonica and M.J. Egoville performed the analysis.

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