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Design of a Single-Stage Mechanical Fat Separator

ABSTRACT: Thermal fractionation of highly saturated fats can address the current nutritional concerns related to the amount, type, and saturated-fat level of lipids in foods. Separation of fats into different melting fractions allows for tailoring natural fats into functional and nutritional groups, while also allowing for the use of the less desirable fractions in nonfood applications. The single-stage laboratory-scale separator described herein separates fats into two gross melting fractions, termed high and low. The latter fractions can be further separated by adjusting the cooling zone temperatures, resulting in secondary fractions with different melt patterns. Thermal analysis of the hard fractions shows a 10–15°C shift to a higher melting point.
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KEY WORDS: Butter oil, design, DSC, fat separation, fractionation, hard fractions, laboratory scale, lard, melt profile, tallow.

Though milk fat enjoys wide acceptance, due to its flavor and textural properties, consumption in the United States has declined in recent years (1). Changing dietary habits have created the need for tailored fats to address nutritional concerns. Milk fat is a significant quality-enhancing ingredient in foods. Milk fat can be made more nutritionally acceptable through selective blending of separated triglycerides. The development of structured lipids, based on the triglyceride chain-length, yields the most flexibility in adapting to future demands (2).

Several methods of crystallization and fractionation, based on triglyceride melting point, have been described (3,4). Functionality in certain products is based on the product's melting point, and a process that modulates melting characteristics would increase the opportunities for increased use of milk fat. High-melting fractions of milk fat are employed in specialty bakery products, and other fractions have been used in confections (5,6).

Milk fat fractionation can be accomplished through chemical, supercritical extraction, distillation, and dry-melt methods (7,8). Dry fractionation is a thermally controlled process in which crystallization is induced, and the fat crystals are filtered. Milk fat crystallization, on an industrial scale, provides several fractions with different melting profiles after several fractionation steps (5). Other hard fats (tallow and lard), with a saturated fat profile similar to butter, have been fractionated in a continuous process. The two-step continuous pilot-plant fractionation process is constrained by size and cost of operation and prevents its use as a small-scale laboratory fractionator (9). This study describes a device that offers a single-step separation of high-fat fractions and investigates its effectiveness for gross separation for butter oil, tallow, and lard.

MATERIALS AND METHODS

Anhydrous butter oil was purchased from a commercial distributor (Land o' Lakes, Minneapolis, MN). Deodorized lard and tallow were received from Holsum Foods (Waukesha, WI).

Fractionation process. The fractionation device is constructed of a cylindrical pipe, 1.52 m in length and 127 mm in diameter, with a 6.35-mm thick inside Teflon coating. The barrel has four heating zones, with independent heating/cooling copper coils wound to cover each zone. A top-mounted motor drives an impeller blade that provides continuous mixing. Thermocouples are attached to each zone, which allows for continuous monitoring of temperatures (Fig. 1). The separation profile is set from top to bottom as follows: Zone 4 = cooling/solidifying zone, temperature determined by the desired product; Zone 3 = preset at 35°C; Zone 2 = preset at 45°C; Zone 1 = preset at 50°C. The melting profile of the fractionated fat is controlled by the temperature in the cooling zone. Three different separation profiles were used for butter oil (B20, B25, and B30), with the different crystallization temperatures for Zone 4 set at 20, 25, and 30°C, respectively. Other fats (tallow and lard) were separated with Zone 4 set at 20°C. Each separation was replicated once. Fat samples to be fractionated were melted at 50°C in batches. The samples were pumped in through the top (Zone 4). After batch crystallization, the separated hard fractions were removed with metal scrapers. The remaining liquid fractions were drained through the bottom drain.

Analysis. A Perkin-Elmer differential scanning calorimeter, Model DSC-7, equipped with an Intra cooler II refrigeration unit (Nitrogen flush; 20 psi), was used to measure thermal characteristics (Perkin-Elmer Corp., Norwalk, CT). Ten mg (±1) of sample was weighed into aluminum pans (Perkin-Elmer) and hermetically sealed. An empty sample pan was used as reference. Samples were heated from −10 to 60°C at

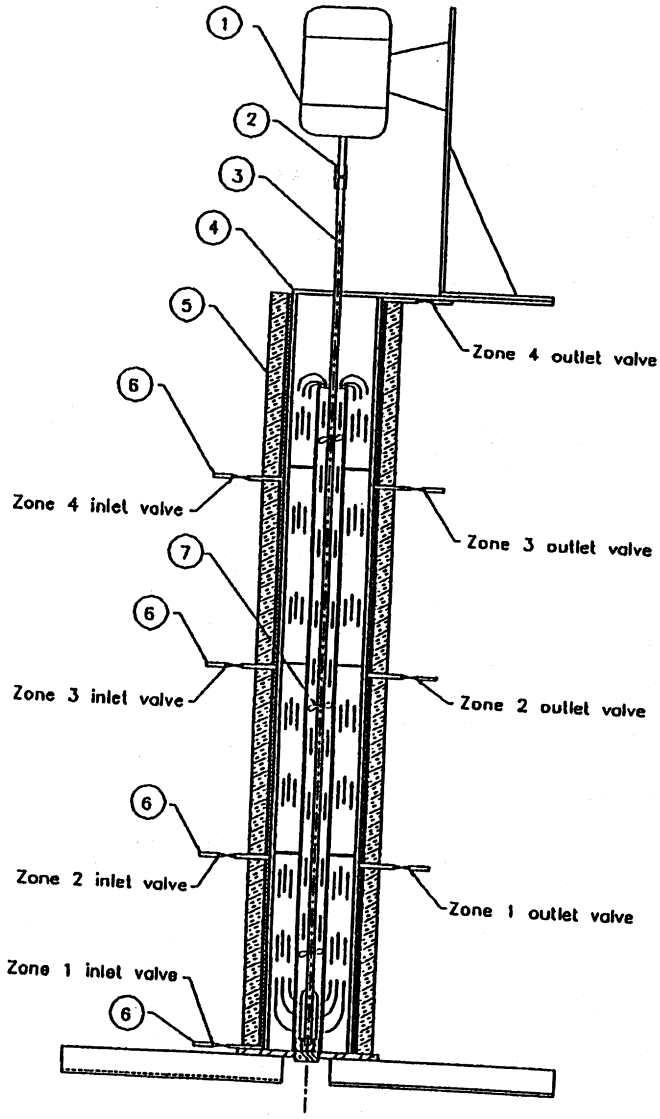

FIG. 1. Diagram of the pilot-scale fat separator: 1: motor (Leland Faraday Model M-01502; Leland Faraday Electric Company, New York); 2: drive coupling; 3: impeller shaft; 4: shell, 5″ IPS, Sch. 40; 5: insulation, fiberglass, 1″ thick, full length; 6: tubing, copper, ¼″; 7: impeller, 3 places.

5°C/min after initial cooling to −15°C at 5°C/min. The heat of melting, in joules per gram of sample, was determined by dividing the area under the curve by sample weight. Melting patterns, areas, and peak melting points were obtained from differential scanning calorimetry (DSC) thermograms.

Triglyceride compositions of the high-melting fat fraction samples were determined by high-performance liquid chromatography (HPLC). The analysis of the hard fraction of butter oil, tallow, and lard in order of separation (0, 24, 48, 72 h) was made at selected separation times. Analyses were performed with a Hewlett-Packard 1090 HPLC system (Hewlett Packard, Inc., Avondale, PA), equipped with an ODS reverse phase column. Samples (25 μL) were manually injected into the HPLC, which was programmed to run for 25 min with a binary solvent of acetone and acetonitrite at a constant flow rate of 1 mL/min, as described elsewhere (10).

RESULTS AND DISCUSSION

The melting pattern for butter oil in the DSC thermogram showed main peaks from 10 to 60°C. The thermograms show the shift of the high melting peak (Fig. 2). The peaks represent butter oil separation profile B20 (Zone 4 at 20°C) for the high-melting fractions. Similar trends were observed for melting profiles B25 and B30 (Table 1). The most significant increase in melt temperature occurred after 24 h, where the melt point increased 10–12°C. Uniform increases in melt points and shifts toward higher temperatures were obtained at certain separation temperature profiles. For most temperature profiles, optimal separation time was at 48 h; rather, the peak melt point decreased with continued holding to 72 h.

The triglyceride compositions of the melt fractions were determined by HPLC. The triglyceride profile of the butter oil fraction at B20 separation is presented in Figure 3 for low-

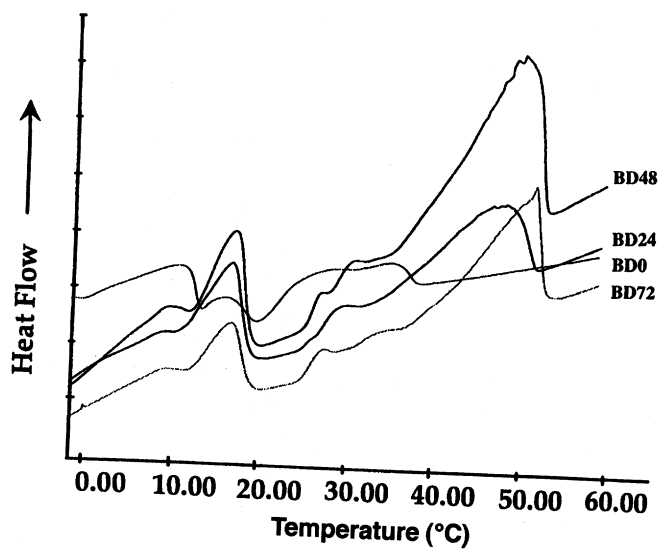

FIG. 2. Shifts in peak melting point of hard fractions of butter oil; BD24: separation time, 24 h.

TABLE 1
Peak Melting Point Profile for High Fractions of Butter Oil[a]

Sample	Peak melting (°C)			
	0 h	24 h	48 h	72 h
B20	38.53 (1.5)	48.33 (0.2)	48.95 (0.9)	51.42 (1.2)
B25	38.84 (1.3)	50.43 (0.8)	50.13 (0.5)	51.67 (0.3)
B30	38.83 (1.4)	50.67 (0.2)	53.17 (1.1)	51.72 (0.3)

[a]B20: Zone 4 Cooling set at 20°C. Numbers in parentheses are standard deviations.

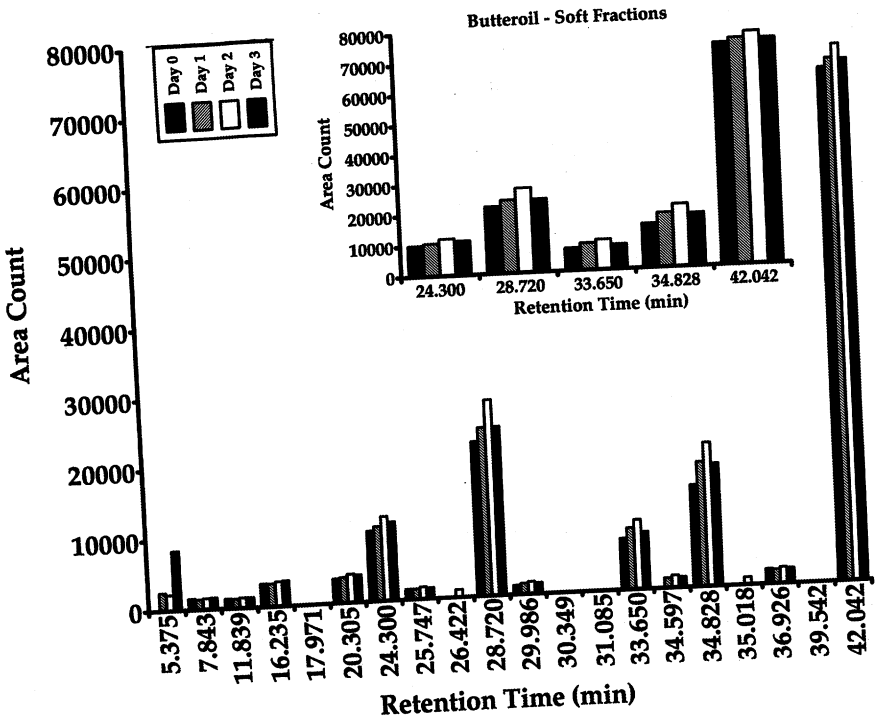

FIG. 3. High-performance liquid chromatography profile of selected triglycerides from butter oil; BH24: Butter oil, soft fraction, 24 h.

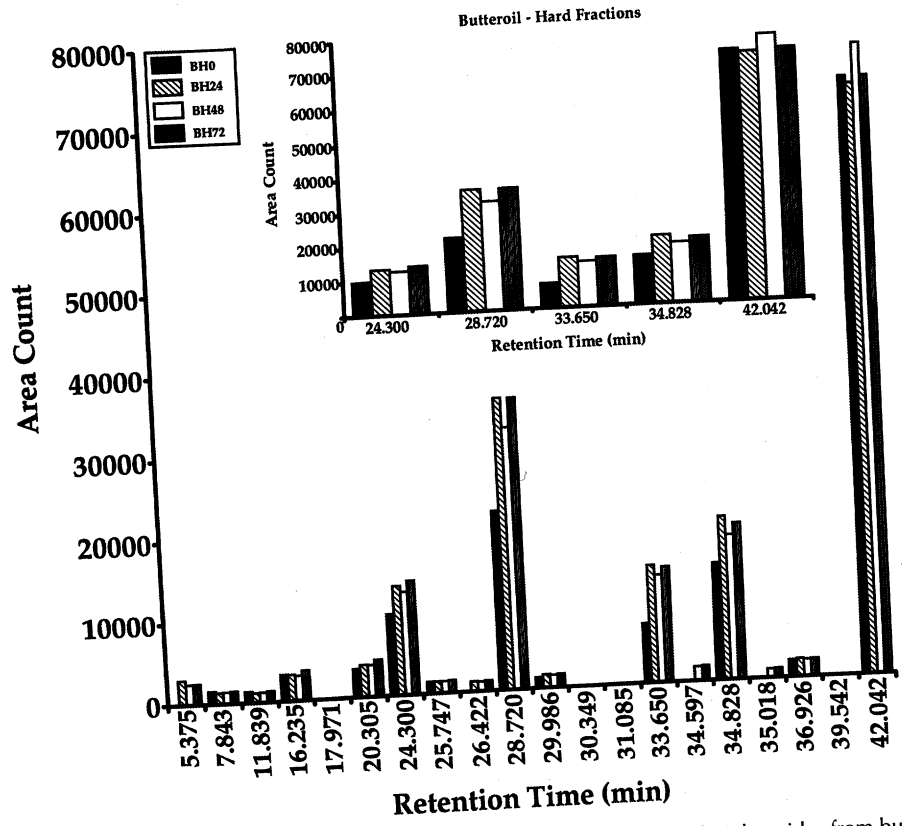

FIG. 4. High-performance liquid chromatography profile of selected triglycerides from butter oil; BH24: Butter oil, hard fractions 24 h.

melting fractions and in Figure 4 for high-melting fractions. The triglyceride distribution varied as the time of crystallization was increased. Significant increases in fractions of triglycerides in the retention times 33–35 min show clear separation of soft and hard fractions, consistent with the observed thermal analysis. Though clean separation between the high- and low-melting fractions is beyond the range of practical application of dry-melt crystallization, increases in the higher-melting fractions for selected triglycerides are evident (see inserts in Figs. 3, 4). Triglyceride chainlength as a function of retention time has been reported (10). HPLC data indicated that separated fats can be fractionated into selected triglyceride compositions. Structured lipids that provide functional benefits can be tailored from milk fat and other fats after successful crystallization (2).

Melt crystallization with the laboratory-scale separator was a function of time and temperature. Slow nucleation and growth of crystals in the melt fraction are normally driven by temperature, time, and agitation (3). Process time is the main factor in crystallization kinetics, although reported time for optimal crystallization has varied from 4 to 48 h, depending on the fractionator size (4).

The solid fat contents of the butter-oil fractions are presented in Figure 5 for the soft fractions and in Figure 6 for the hard fractions. The profiles followed reported patterns for solid fat content (4). The higher-melting fractions (Fig. 6) showed more solid fat content than the lower-melting fractions. This agrees with the HPLC analysis and is in agreement with trends reported in the literature (4).

Structured lipids based on triglyceride chainlength are classified into short- and medium-chain (C_4–C_{10}), and long-chain (C_{14}–C_{18}) triglycerides. Our process separates fats into low- and high-melting fractions; the medium-chain triglycerides predominate in the low-melting fractions, while the long-chain triglycerides are more predominant in the high-melting fractions. Functional advantages of the high-melting fractions of milk fat are best realized in bakery and pastry applications, where crystallized milk fat is suitable for firm products (6).

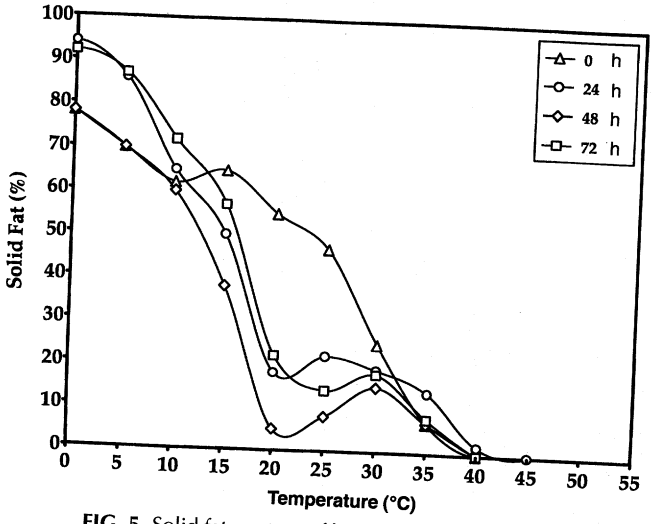

FIG. 5. Solid fat content of butter oil, soft fractions.

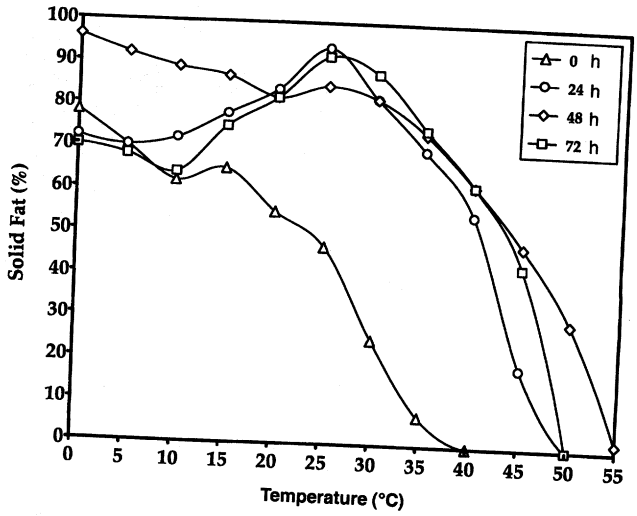

FIG. 6. Solid fat content of butter oil, hard fractions.

Shifts in the melting profile of the high-melting fractions of lard and tallow are presented in Table 2. Melt point temperatures increased approximately 10–15°C; similar trends in the shift of melting characteristics were observed.

The HPLC analysis of the lard and tallow fractions shows shifts in triglyceride profiles toward the higher-melting triglycerides, similar to those observed with milk fat (Figs. 7 and 8). Wang and Lin (11) have shown that lard can be crystallized and fractionated to provide an edible plastic fat that is suitable for shortening or hardening agents. A two-step process has been described (9) for separating confectionery fat from tallow for use as a cocoa butter replacer.

Functional properties and nutritional quality are the two main driving forces in fat crystallization and separation (4). Identifying a particular triglyceride molecular species and concentrating it to increase yield should increase the use of fats that are now being rejected for particular nutritional defects. This study is the first step in the development of a laboratory-scale selective fat fractionator. By adjusting the separator temperature profile, desirable triglyceride fractions, such as the low- and high-melting fractions described here, can be obtained. The fractionation is not "sharp" because some lower-melting fraction peaks are present in the desired prod-

TABLE 2
Peak Melting Point Profile for High Fractions of Separated Lard and Tallow[a]

Sample	Peak melting (°C)			
	0 h	24 h	48 h	72 h
Lard	41.52 (0.8)	53.15 (0.7)	57.54 (0.3)	55.26 (0.6)
Tallow	44.39 (2.0)	54.52 (0.6)	54.89 (0.9)	55.65 (0.5)

[a]Numbers in parentheses are standard deviations.

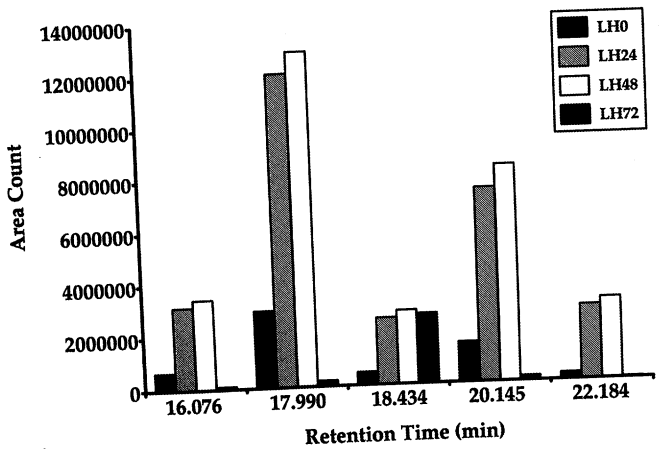

FIG. 7. High-performance liquid chromatography profile of selected triglycerides from lard; LH24: lard, hard fraction, 24 h.

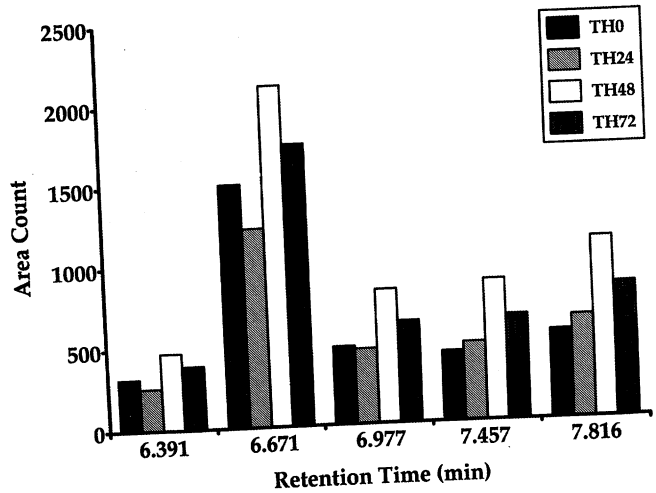

FIG. 8. High-performance liquid chromatography profile of selected triglycerides from tallow; TH24: tallow, hard fraction, 24 h.

uct fraction (Fig. 2); however, the simplicity of the design allows for ease of construction and use. The single-step separator permits pilot-scale crystallization of fats. The melt profile or peak melt point can be adjusted, based on the selection of cooling temperature in Zone 4. Particular triglyceride compositions can then be concentrated through selective crystallization.

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