

# Physical Properties of Encapsulated Spray-Dried Milkfat

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## ABSTRACT

Spray-dried encapsulated milkfat powders were prepared from stable emulsions containing 40–60% milkfat and carbohydrate matrices. Moisture content of the spray-dried powders varied from 1–4%. Lowest free fat content (< 10%) was found in powders with 40% fat, encapsulated in sucrose. Angles of repose ranged from 37 to 46°, and correlated with powder flow ( $p = 0.01$ ). Bulk density was dependent on the encapsulant and declined with increasing fat content. Product density did not influence powder recovery through the cyclone of the dryer. Particle size distribution ranged from 20 to 120  $\mu\text{m}$  with 80% of the particles < 100  $\mu\text{m}$ . Powders with best physical properties were made with 40–50% butteroil encapsulated in sucrose.

Key Words: milkfat, spray drying, encapsulation, sucrose, butter oil

## INTRODUCTION

REDUCED FAT consumption due to changing dietary habits has resulted in a worldwide surplus of butter. This trend is expected to continue and will create severe storage problems (Kehagis and Radema, 1973; Anonymous, 1989; USDA, 1991). Salted butter can be readily stored frozen for up to 3 yrs, but freezer space is limited and costly. A storage life of 12–24 mo at ambient temperatures can be achieved when the milkfat is converted to a powder (Claypool, 1984). Although production of high-fat butter powders is technologically possible, widespread use has not followed, due to processing difficulties (Hansen, 1963; Prasad and Gupta, 1979; Patel et al., 1987). Spray-drying of butter with functional encapsulants such as starch, maltodextrin, or gums would enhance stability of the dry powder if microcapsules formed would protect the milkfat from oxidative deterioration during storage. Such powders may easily be recombined or incorporated as ingredients into many food systems (Frede and Ehlers, 1991; de Man, 1984).

The choice of encapsulant is critical as the material will influence emulsion stability before drying, flowability, mechanical stability and shelf life after drying. Superior emulsifying capacity and oil retention have been reported for some materials. For example, flavorings and citrus oils have been encapsulated in food gums and modified starches that behave like gums (Tripp et al., 1971; Bangs and Reineccius, 1990). For the production of butter powders, the solids-not-fat matrix may consist of milk protein products such as nonfat dry milk, sodium caseinate or whey proteins, various sugars, starches, gums, emulsifying agents and/or sodium citrate (Frede and Ehlers, 1991). Sodium caseinate has been reported to be the most effective emulsion stabilizer; it has also been recommended that the size of the fat globules in the emulsion be small (< 0.6  $\mu\text{m}$ ) (Frede and Ehlers, 1991). Powder stickiness and lumpiness are directly related to emulsion stability; the more stable the emulsion the more free-flowing is the powder.

Most published information refers to preparation of butter powders with > 75% fat. Our objective was to investigate the physical characteristics of spray-dried powders containing 40 to 60% milkfat, prepared with varying encapsulants.

## MATERIALS & METHODS

ANHYDROUS BUTTEROIL was purchased from a commercial source (Land O' Lakes, Minneapolis, MN). Cream was obtained from a local dairy (Longacre's Modern Dairy, Inc., Barto, PA). Encapsulants were sucrose (Domino's, Domino Sugar Corp., New York, NY); N-starch, all-purpose flour (ADM Milling Co., Kansas City, MO); and M-starch, modified starch (Capsul<sup>®</sup>, National Starch and Chemical Co., Bridgewater, NJ). An emulsifying agent, comprised of mono- and di-glycerides (American Ingredients Co., Kansas City, MO), was also used. Nonfat dry milk served as protein source (Maryland & Virginia Milk Producers Association, Inc., Laurel, MD).

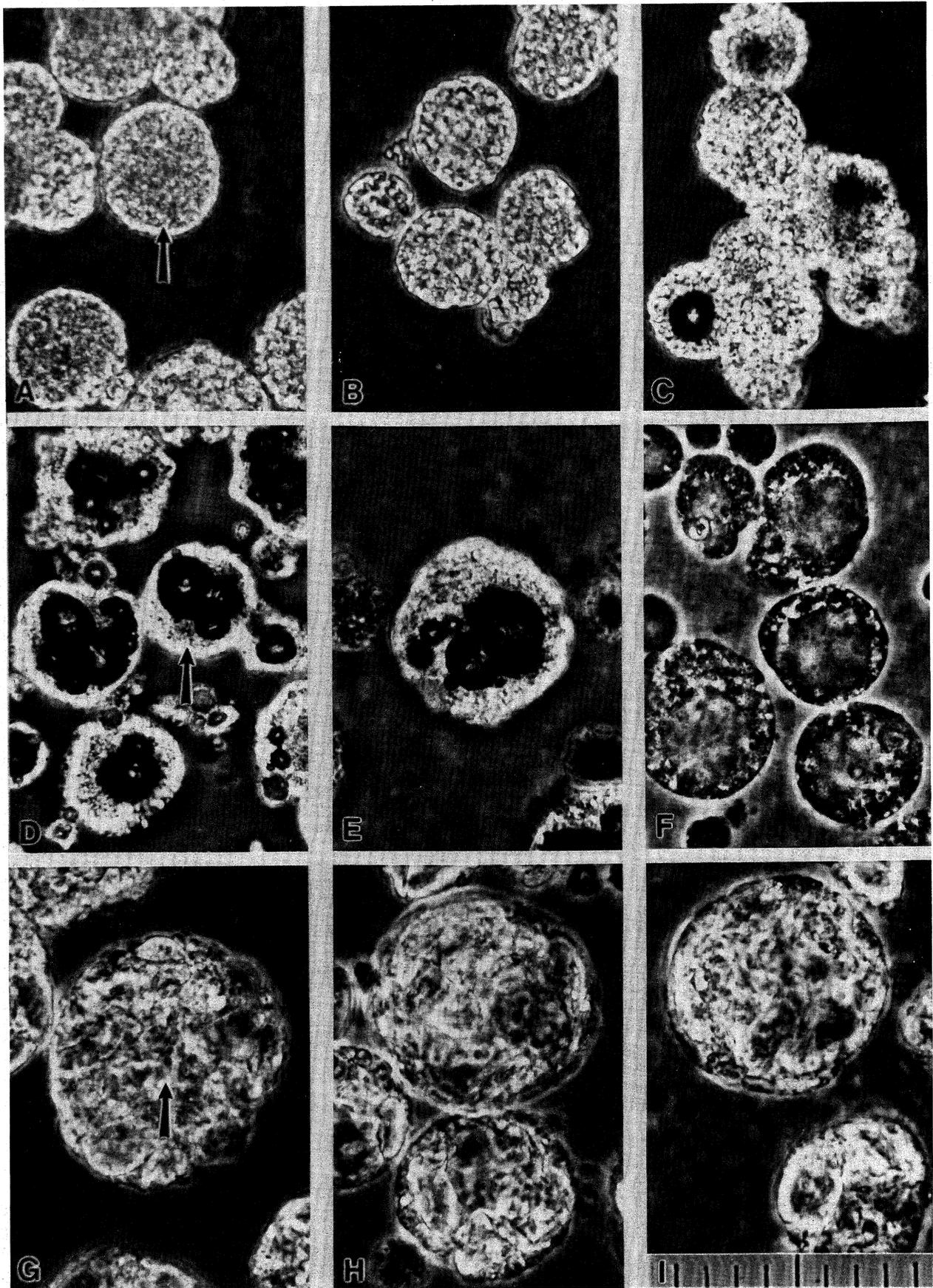
Sample preparation was carried out by following a full  $2 \times 3 \times 3$  factorial design, completely randomized and replicated. Two forms of milkfat, anhydrous butteroil or cream (40% fat) were emulsified at three fat levels, 40, 50, or 60%, with three encapsulants, sucrose, unmodified starch (all-purpose flour), or modified starch, added emulsifier and nonfat dry milk.

Encapsulated powders were prepared with 40–60% milkfat, 5% emulsifier, and 5% skim milk powder. The processing sequence was as follows: The encapsulant of choice was dry-blended with nonfat dry milk solids. The blended mixture was dispersed in water to form a pasty slurry of  $\approx 25\%$  total solids. The anhydrous butteroil or cream, and the emulsifier were heated to 23.9°C. The two blends were combined (40% total solids) and mixed for 5 min with a milk stirrer, after which the slurry temperature was slowly increased to 62.8°C with constant stirring, and homogenized at 17.2 MPa with a Manton-Gaulin Triplex homogenizer (Model 100 DJF3855, APV Gaulin, Inc., Everett, MA), followed by spray-drying in a compact dryer (APV Crepaco, Inc., Attleboro Falls, MA) with inlet temperature 193.3–196.1°C and outlet temperature 82.2–87.8°C. Powders were produced batch-wise, removed from the dryer after 30 min and stored at 4°C. When unmodified starch (all-purpose flour) was used as encapsulant, it was necessary to homogenize at 10.3 MPa and 54.4°C to accommodate its pasting properties. The milk protein content ranged from 2% for powders made with butteroil to 4% for powders made with cream as the fat source.

Particle structures of the microcapsules were evaluated with an optical microscope equipped with optics for phase contrast (Olympus microscope, model BH2; Olympus Corp., Lake Success, NY). Moisture was determined by an AOAC (1984) method by drying under vacuum for 4 hr at 102°C. Extractable fat was determined by dispersing 10g powder in 50 mL carbon tetrachloride and shaking for 15 min (Anonymous, 1978). The soluble fraction was filtered and the solvent was evaporated, leaving the fat. Extractable fat was expressed as the fat recovered from the powder, divided by 10. Efficiency of encapsulation represented the difficulty in extraction of residual fat. Bulk densities of all powders were determined by dividing the weight of powder (g) contained in a 200-mL stainless steel cylinder (A/S Niro Atomizer, Copenhagen, Denmark) by its volume in  $\text{cm}^3$ . Packed bulk densities were calculated from the weight of powder contained in the cylinder after being tapped 100 times. Density measurements ( $\text{g}/\text{cm}^3$ ) were done in triplicate.

Flow characteristics were evaluated by permitting 80g of powder to flow through a funnel to form a heap; angle of repose was calculated as  $\theta = \cotan h/r$  from the dimensions of the pile where  $h$  = height of the powder pile and  $r$  = radius of the base of the pile. The angle of repose is defined as the base angle formed when a given weight of powder flows through a funnel of known dimensions to form a pile (Sjollema, 1963). The flow behavior of nonfat dry milk was used as a control. The relative flow of the powder with time was measured by permitting 80g to flow through funnels of outlet diameters 1.27–2.54 cm with gentle shaking (FMC/Synthron, Homer, PA). Time of flow was recorded. Relative flow rate was calculated as powder weight (g) divided by time (sec).

Particle size distribution was estimated by passing 100 g of each powder through a series of sieves with screen openings ranging from



**Fig. 1**—Optical phase contrast micrographs of microcapsules of butteroil in carbohydrate matrices. Sucrose with 40% (A), 50% (B), and 60% (C) butteroil. M-Starch with 40% (D), 50% (E), and 60% (F) butteroil. N-Starch with 40% (G), 50% (H), and 60% (I) butteroil. Replica line grating of 10 micrometers (I). Arrows on particles on 40% butteroil samples point to highly refractile capsules of sucrose (A), M-Starch (D), and to the matrix of a particle containing N-Starch (G).

**Table 1—Moisture content of spray-dried powders<sup>a</sup>**

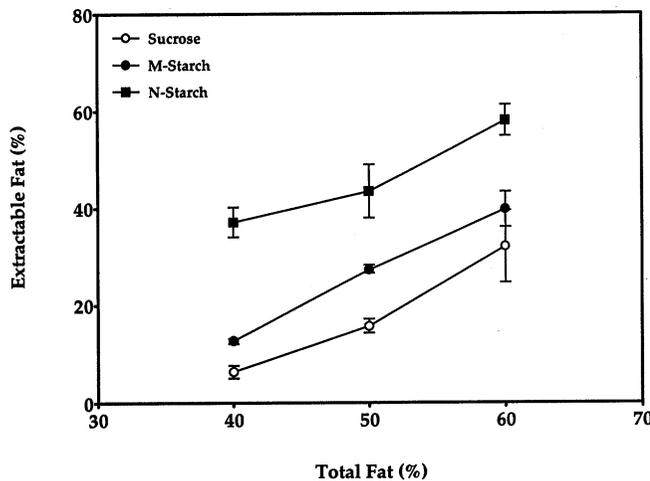
Wall material Milkfat	Sucrose		M-Starch		N-Starch	
	BO	Cream	BO	Cream	BO	Cream
	% moisture					
40	1.97	1.24	1.44	1.61	3.10	3.20
SD	0.7	0.2	0.2	0.1	0.6	0.3
50	1.61	1.62	1.21	1.04	3.76	1.13
SD	0.2	0.2	0.4	0.1	0.5	0.1
60	1.19	1.10	1.00	1.76	1.22	1.00
SD	0.6	0.4	0.3	0.3	0.1	0.2

<sup>a</sup> Moisture on wet basis. M-Starch = Modified starch. N-Starch = All-purpose flour. BO = Butteroil. SD = standard deviation.

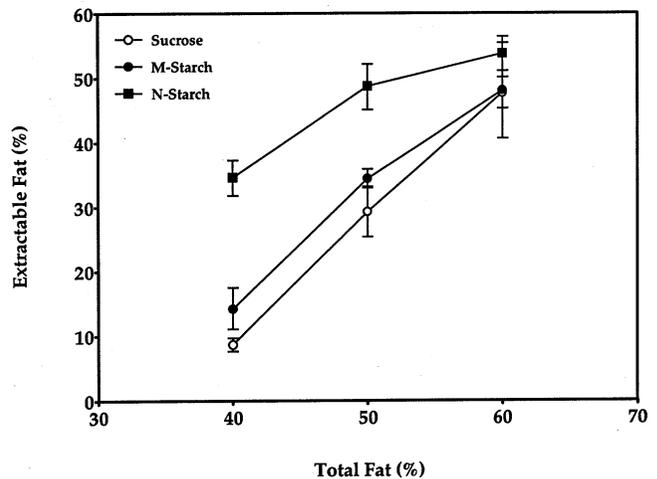
**Table 2—Flow properties of spray-dried powders<sup>a</sup>**

Wall material Milkfat	Sucrose		M-Starch		N-Starch	
	BO	Cream	BO	Cream	BO	Cream
	g/sec					
40	5.71	1.78	0.18	0.27	0.27	0.34
SD	0.01	0.51	0.08	0.06	0.03	0.12
50	4.21	0.36	0.20	0.28	0.45	0.52
SD	0.40	0.14	0.04	0.14	0.08	0.26
60	1.15	0.23	0.29	1.07	0.99	0.92
SD	0.28	0.05	0.09	0.61	0.22	0.55

<sup>a</sup> Flow Rate. M-Starch = Modified starch. N-Starch = All-purpose flour. BO = Butteroil. SD = standard deviation. Moisture content of powders was not adjusted.



**Fig. 2—Effect of encapsulant on percentage of extractable fat. Source of fat: Anhydrous butteroil.**



**Fig. 3—Effect of encapsulant on percentage of extractable fat. Source of fat: Cream.**

100–500  $\mu$ m. The stack was shaken and tapped with a Rotap<sup>®</sup> shaker (Tyler Co., Cleveland, OH) for 5 min. Powder distribution by weight was recorded and cohesiveness was estimated by the percentage of powder that aggregated or did not pass through the 500-micrometer sieve. Results were analyzed for trends with the Statistical Analysis System of General Linear Models (SAS Institute, Inc., 1991).

## RESULTS & DISCUSSION

THE MAIN PURPOSE of preparing spray-dried fats is to enhance their handling properties, for example, in storage, transport and blending with nonfat ingredients. Milkfat must be powdered by using a carrier because it contains appreciable amounts of low-melting triglycerides. As the fat content increases, the choice of carrier constituents becomes more critical. Our studies were in the medium fat range, not only for greater ease in processing, but also because we believed reduced-fat products are more desirable as ingredients in “light” processed foods of reduced calorie content.

The particle structures of the various microcapsules were observed (Fig. 1). The structures were distinct and dependent on the carbohydrate matrix. Mean particle size as measured by image analysis, increased as amount of encapsulated milkfat increased (Onwulata et al., 1993). Clustering of particles was the result of surface milkfat on the particles, which was influenced by the ratio of encapsulated milkfat to encapsulant. The flow properties correlated with cohesiveness and extractable fat.

Moisture content of the spray-dried powders varied from 1–4% (Table 1), with highest moisture in the sample prepared with unmodified starch and anhydrous butteroil. The amount of fat and the type of encapsulant in the emulsion significantly affected moisture content. The affinity for water was largely dependent on encapsulant, but no powder had a moisture content > 4%. Nonfat dry milk and whole milk powders have

moisture contents ranging from 2 to 4%. The moisture content is critical in dehydrated products, because a small residue of water appears to be a major factor in inhibiting fat oxidation (Koch, 1962). No optimum moisture content for butter powders has been indicated.

The efficiency of the encapsulants in successfully encapsulating milkfat was examined by shaking the powders with carbon tetrachloride for a predetermined time to measure the amount of fat extracted (Fig. 2 and 3). Extracted fat depended on type of encapsulant, source of the milkfat, and fat content. In terms of extractable fat, butteroil appeared to form better capsules than did cream (Fig. 2). The efficiency of encapsulation decreased as fat content increased in both cases. The efficiency of various matrices in encapsulating fat has been reported as high as 94% with fat contents  $\leq$  20% (King, 1976; Jackson and Lee, 1991). Whey proteins have been used as an encapsulant with varied efficiency, depending on fat content (Young et al., 1992); the encapsulation efficiency decreased as fat content exceeded 25%. In our study, powders with sucrose encapsulant showed much higher fat retention (94%, 40% milkfat, anhydrous butteroil as fat source, Fig. 2) with a much more rigorous extraction process. Powders with M-starch were 85% efficient while N-starch was the least efficient (62% with a 40% fat content (Fig. 2). When cream was the fat source, encapsulation efficiency showed a significantly greater decline in all cases when fat content  $\geq$  50% (Fig. 3). These results suggest that the emulsion formed before drying was not as stable. That is, fat globule size was larger and droplets were less dispersed regardless of the additional emulsifying capacity contributed by the presence of phospholipids in the cream.

Bulk densities of the spray-dried powders were dependent on the encapsulant (Fig. 4 and 5). Highest bulk densities were found in powders made with N-starch and anhydrous butteroil (Fig. 4). Densities of powders containing sucrose or modified starch were not significantly different; however, there was an

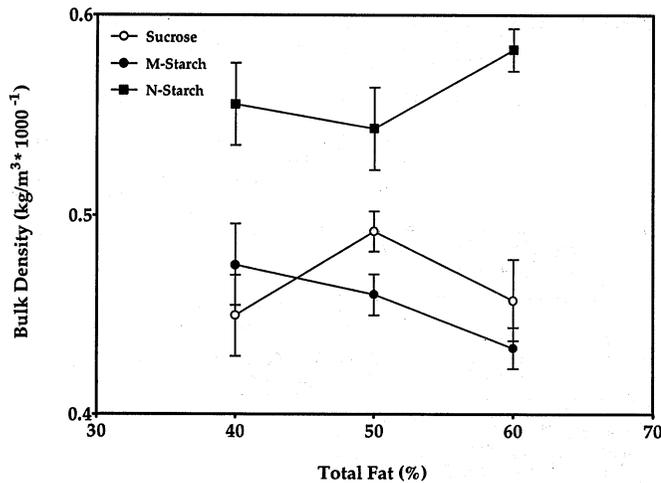


Fig. 4—Effect of encapsulant on bulk density of powders with different fat content. Source of fat: Anhydrous butteroil.

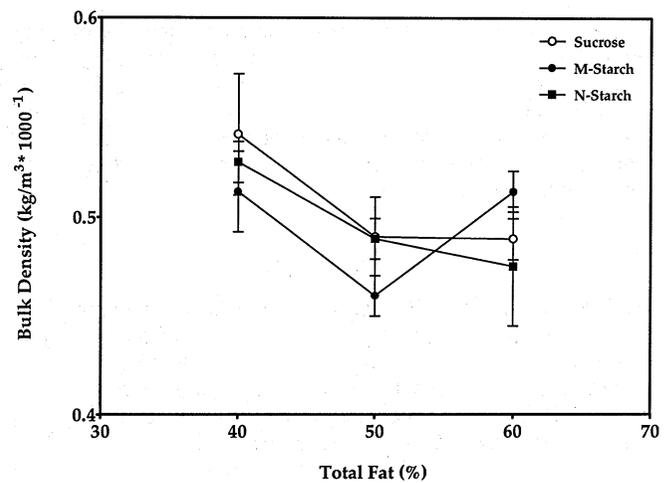


Fig. 5—Effect of encapsulant on bulk density of powders with different fat content. Source of fat: Cream.

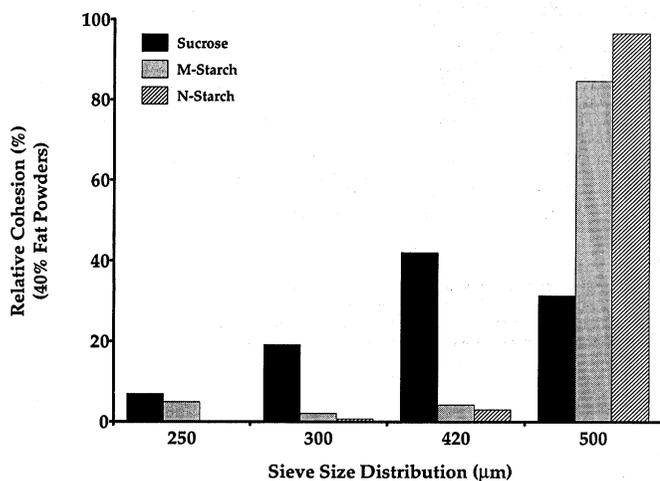


Fig. 6—Effect of encapsulant on particle size distribution and cohesiveness of powders with 40–60% fat. Source of fat: Anhydrous butteroil.

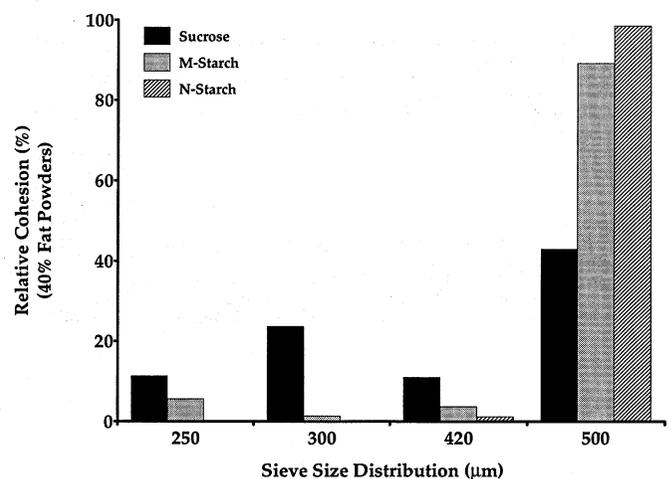


Fig. 7—Effect of encapsulant on particle size distribution and cohesiveness of powders with 40–60% fat. Source of fat: Cream.

Table 3—Angle of repose of spray-dried powders\*

Wall material	Milkfat	Sucrose		M-Starch		N-Starch	
		BO	Cream	BO	Cream	BO	Cream
		(Degrees)					
40		45.6	40.2	39.7	42.3	38.4	38.0
	SD	1.2	0.7	2.3	2.2	1.2	1.6
50		38.0	38.9	40.9	42.0	38.5	41.5
	SD	1.4	0.8	1.4	0.7	1.7	1.6
60		40.8	37.8	42.0	39.9	40.5	40.5
	SD	4.9	2.3	1.9	3.6	2.6	1.8

\* Angle of repose. M-Starch = Modified starch. N-Starch = All-purpose flour. BO = Butteroil. SD = standard deviations.

apparent difference in bulk density in those powders with unmodified starch. Product bulk density did not influence powder recovery through the cyclone of the dryer. A decrease in bulk density was observed for most powders regardless of fat source, when fat content increased to 50%. Occlusion of air within the microcapsules was determined by microscopic examination. Inclusion of air bubbles in the particles lowered bulk density. Particle density is important during transport and storage because the particles may become compacted and form large lumps.

Measurement of powder flow, compared to the flow of nonfat dry milk at 3.0 g sec<sup>-1</sup>, showed that powders with sucrose and anhydrous butteroil were more free-flowing than the other

samples (Table 2). As fat content of the powder increased, flowability decreased sharply. Flowability was also influenced by the ratio of encapsulated to extractable fat. Powders with higher levels of unencapsulated fat on the surface (therefore, a greater amount of extractable fat) tended to stick together and form lumps, which impeded flow.

An average angle of repose has been reported for nonfat dry milk (43°) (Sjollem, 1963). Angles of repose for the powders ranged from 37° to 46° with a mean angle of 40° (Table 3). It has been suggested that powders with angles of repose < 35° should be considered to be free-flowing, those with angles 35° ≤ 45° as cohesive, while powders with angles of repose > 55° had little or no flow (Sjollem, 1963). Relative to the angle of repose of nonfat dry milk, our completely encapsulated milkfat powders (sucrose encapsulant) could be considered free-flowing; the stickiness arises from unencapsulated fat on the surface of the powder particles (Peleg et al., 1973; Peleg, 1983). Powders incorporating high-fat levels have flow properties different from those of other food powders. Our powders, with angles of repose 37° ≤ 42° were relatively free-flowing regardless of particle aggregation due to unencapsulated fat. Angle of repose significantly correlated with powder flowability (ρ = 0.01).

The particle distribution for powders with butteroil or cream showed a distribution range of 20 to 120 µm, with 80% of the particles < 100 µm in diameter (Onwulata et al., 1993). The powders were cohesive, forming large aggregates, their number increasing with increasing fat content (Fig. 6 and 7). Su-

crose-containing powders were relatively free-flowing and nonlumpy, compared to the other powders. Particle clusters were caused by large amounts of unencapsulated fat that acted as a binder, increasing cohesiveness. Sieve size distribution was directly related to fat content of the powders; as percent fat increased, the unencapsulated fat increased and cohesiveness (stickiness) increased.

### CONCLUSIONS

RELATIVELY FREE-FLOWING powders containing 40–60% milkfat were successfully spray-dried, with a variety of encapsulants. The best powders contained sucrose as encapsulant with anhydrous butteroil as fat source; extractable fat was < 10% in those powders. Such products can be readily used as food ingredients in processed foods where sweetness is required. However, their long-term storage stability remains to be determined.

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