Chapter 18

Development of Novel Ingredients from Surplus Milkfat

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In the U.S., fat consumption patterns changed in the mid 1980's, resulting in surplus butter inventory. The management and long-term storage of surplus butter is cost prohibitive because of the extremely low refrigeration temperature required. A cost saving alternate is to dry the milkfat with functional encapsulants such as starch, proteins or gums, making the powders shelf-stable at ambient temperature. By using microencapsulation techniques, the milkfat is entrapped within matrices formed by protein and carbohydrates that may protect the fat from oxidative deterioration during storage. Spray drying this encapsulated milkfat results in free-flowing powders that are shelf stable at ambient temperatures, and reduces the need for expensive frozen storage for milkfat. Substituting powders prepared with all-purpose flour and 50% milkfat for shortening in baking trials increased volume with minimal effect on textural quality, demonstrating their suitability as bakery ingredients.

Reduction of fat in the diet in the United States has led to a surplus butter supply, despite the desirable flavor and textural properties that butter provides. As this trend is expected to continue, a more efficient means of long term storage for butter is needed. Milkfat is readily susceptible to autoxidation and becomes rancid at ambient or refrigeration temperatures. Salted butter maintains good storage stability when stored frozen for up to three years, but frozen storage is expensive. Transforming surplus butter into a dried form, so that it can be stored for 12-24 mo at ambient temperatures has the potential for increasing the use of milkfat (*1*). It has been shown that spray drying milkfat with functional encapsulants such as starch or other carbohydrates to form free-flowing powders can reduce storage costs and enhance stability by forming fat-containing microcapsules during the drying process to protect the milkfat from oxidative deterioration during storage (*2,3*). Anhydrous butter oil or cream has been successfully encapsulated in carbohydrate matrices (sucrose, all-purpose flour or modified starch). Physical and structural properties varied with the source of milkfat and the type of encapsulant used (*4*). Amorphous glass entraps flavor compounds and protects encapsulated materials from oxidation; however, encapsulant crystallization might result in higher diffusion rates or cause complete release of encapsulated compounds through capsule rupture (*5,6*). Formation of microcapsules is material dependent; for example, oligosaccharides may form multi-component micro-chambers within a capsule (*7*). Formation of microcapsules during spray drying improved physical properties and retained high levels of milkfat (*4,7*). Microencapsulation of milkfat fractions in functional matrices provides new avenues for their use in food formulation (*8*). As an example, the common disaccharides lactose, maltose and sucrose have different solubility and sweetness, suggesting that milkfat encapsulated in these sugars might be good as ingredients in assorted confectionary products (*9-11*). Maltose was shown to retain volatiles within micro-regions through molecular associations (*12*). Remarkably, there is high volatile retention in such powders through the formation of impermeable surface membranes, increased resistance to diffusion at low water content, and the formation of inclusion complexes (*11*). The use of other functional encapsulants such as starch, maltodextrin or gums for the production of spray dried butter powder enhances the handling characteristics and stability of the dry powders and provides protection from oxidative deterioration during storage; such powders offer new avenues for the use of milkfat as an ingredient in food formulation as they can be readily incorporated in food systems (*13*). Their practical utility as ingredients depends heavily on their flow and compaction properties. The choice of encapsulant is critical as it will affect emulsion stability before drying, flowability, mechanical stability and shelf life after drying.

Methodology

Sample Preparation. Encapsulated powders were formulated to have 400, 500 or 600 g/kg milkfat, 20 to 30 g/kg emulsifier, 100 g/kg nonfat dry milk and the remainder, carbohydrate, after drying (*4*). An ingredient list and formulations for powders containing 400 g/kg milkfat are presented in **Table I**. The processing sequence was as follows: The encapsulant of choice was mixed with nonfat dry milk and dispersed in water to make a slurry with approximately 300 g/kg total solids. The anhydrous butteroil or cream, and the emulsifier were heated to 24°C. The two blends were combined to make a 400 g/kg total solids mixture, agitated for 5 min, after which the slurry temperature was slowly raised to 63°C with constant stirring, and homogenized at 17.2 MPa. The homogenized slurry was spray dried in a compact dryer (APV Crepaco, Inc., Attleboro Falls, MA) at an inlet temperature of 193 to 196°C and an outlet temperature of 82 to 88°C. Powders were produced batch-wise and stored at 4°C until used. When all-purpose flour was used as the encapsulant, it was necessary to homogenize at 10.3 MPa and 54°C to accommodate its pasting tendency. The milk protein content ranged from 20 g/kg for butteroil powders to

40 g/kg for cream powders. Emulsifier concentration was based on results showing that higher levels improved baking performance of butter powders (*14*).

Table I.
Ingredient Composition and Sample Formulation for Encapsulated Powders

Ingredients	Moisture g/100 g	Amount needed for powder with 400 g/kg milkfat (g/kg)
Anhydrous Butteroil	0.05	191
Cream (400 g/kg milkfat)	---	436
Sucrose	0.02	246
All Purpose Flour	14.0	284
Modified Starch[a]	5.0	255
Nonfat Dry Milk	3.0 - 4.0	23
Mono- + Di-glyceride	0.05	9
Water	---	627

[a]Capsul® (National Starch and Chemical Co., Bridgewater, NJ).

Process & Product Evaluation

Extractable Fat. The ability of the carbohydrate matrices to retain milkfat was determined (*4*). Extractable fat is expressed as the amount of fat recovered from the powder. The percent fat retained (ϵ) is as follows:

$$\varepsilon = \frac{(\kappa - \lambda)}{\kappa} * 100$$

where κ = total amount of fat in the powder and λ = amount of fat extracted.

The amount of extractable fat varied depending on the type and amount of encapsulant and amount of milkfat (*4*). The relative efficiency of milkfat retention within the carbohydrate matrix for anhydrous butteroil is shown in **Figure 1.** In terms of fat retention, sucrose was clearly the most efficient encapsulant (98 to 96% fat retained). Retention declined for maltose powders (98 to 91%); lactose (97-86%); modified corn starch (86-58%) and the least efficient encapsulant was all-purpose flour (66-48%). Retention declined sharply as fat level increased from 40 to 60%. Most effective encapsulation of both milkfat sources was with sucrose at all fat levels; however, all extracted powders retained at least 40 g/100 g of the total fat even at high fat content (600 g/kg). High fat retention in other matrices has been reported (*15*). Fatty acids tend to bind tightly to proteins of the milkfat globule membrane by covalent linkages (*16*). Strongly polar interactions between milk proteins and milk lipids through hydrogen bonds and electrostatic attraction have been reported during emulsion formation (*17*). It is also known that there is potential for disaccharides to form backbones for complexes (*7*). These protein-lipid-saccharide complexes were resistant to lipid extraction under our experimental conditions.

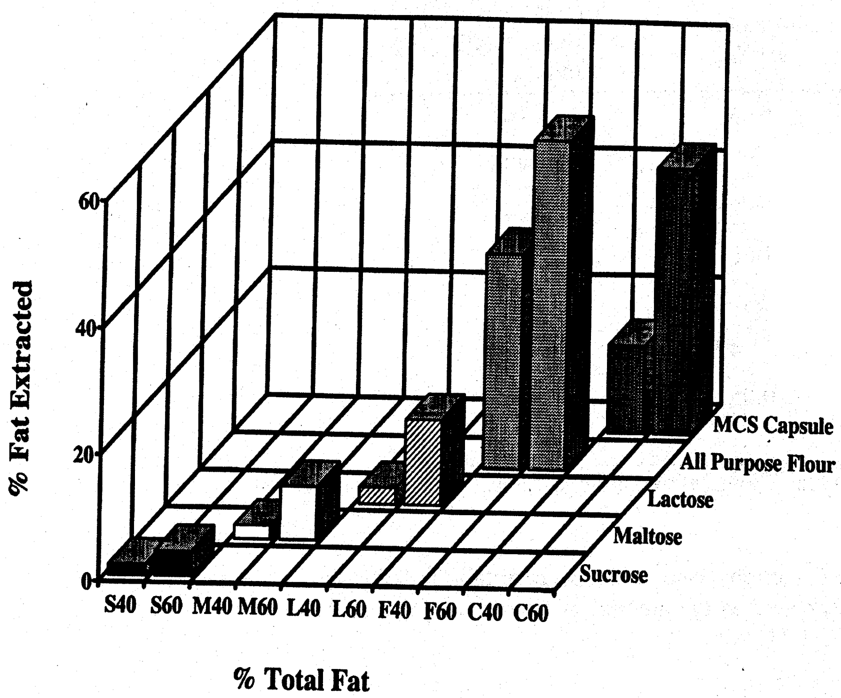

Figure 1.
Amount of fat extracted from encapsulated powders. S40 and S60 = sucrose with 40 or 60% milkfat; M40 and M60 = maltose with 40 or 60% milkfat; L40 and L60 = lactose with 40 or 60% milkfat; F40 and F60 = all-purpose flour with 40 or 60% milkfat; C40 and C60 = modified cornstarch with 40 or 60% milkfat; MCS capsule = modified corn starch capsule.

Particle Size Distribution. Particle size distributions were calculated from sets of optical photomicrographs on microscope slides. Optical magnifications were calibrated using a slide micrometer with an Olympus BH2 phase contrast optical microscope (Olympus Corp., Lake Success, NY). High contrast outlines of the circumferences of 250 particles were traced onto transparent overlays of photographic prints and digitized. Particle diameters were then calculated from projections of the digitized images of the integrated particle areas with a digital image analysis system (Dapple Microsystems, Sunnyvale, CA) running Imageplus software. The diameter was estimated as the best fit of an integrated circular area over an equivalent area of an irregular powder particle. Particle size distributions of the milkfat powder particles encapsulated in all-purpose flour, modified corn starch or sucrose with 400 g/kg butteroil (**Figure 2**) show normal distribution patterns ranging from 20 to 120 μm, with the greatest number of particles around 45 to 55 μm in diameter. Size distributions for 400 g/kg fat-containing powders made from anhydrous

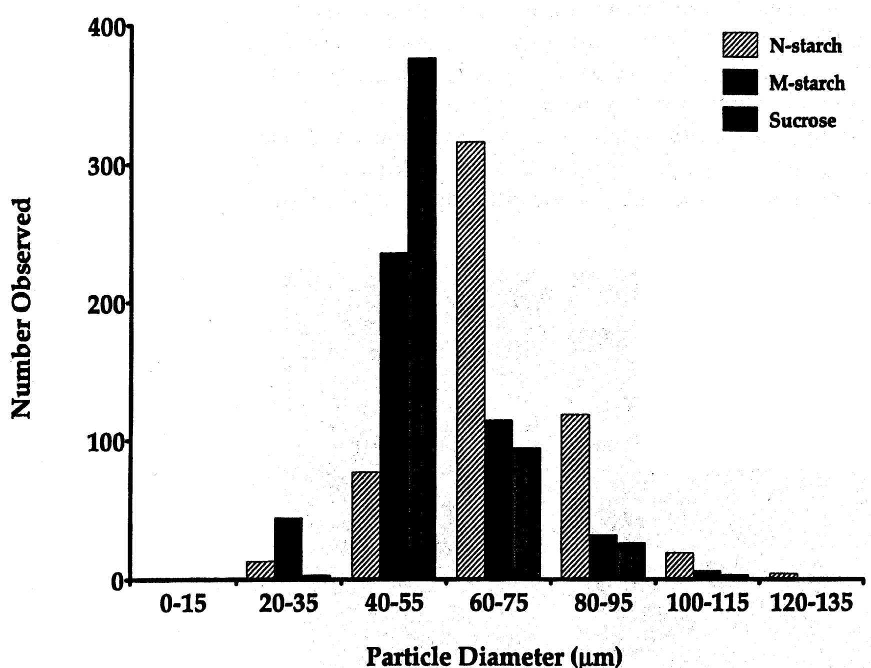

Figure 2.
Frequency distributions of powder particles prepared with all-purpose flour (N-starch), sucrose or modified corn starch (M-starch) as encapsulating agent and containing 400 g/kg anhydrous butteroil as the source of milkfat.

butteroil or cream were similar. There were a greater number of particles of 40 to 55 μm in diameter and a lower number of particles of 60 to 75 μm in diameter for powders made with cream (data not shown). Powders with uniformly sized particles are desirable because they have consistent flow and characteristic functionality. Larger particles, although more easily reconstituted, may be less shelf stable if air diffuses more readily through the voids they contain. Generally more porous structures allow increased oxygen diffusion, increased extractable fat and lower fracture resistance of the particle structure.

Scanning Electron Microscopy. Samples of powders were sprinkled on aluminum specimen stubs coated with Spot-o-glue labels (Avery, Azusa, CA). Observations and images were made in the secondary electron imaging mode of a JEOL Model 840A electron microscope (JEOL, USA, Peabody, MA). Specimen stubs were coated with a thin layer of gold in a DC cold sputtering module in an E306A vacuum evaporator (Edwards High Vacuum, Inc., Grand Island, NY). The powder showed distinct surface features for particles of each encapsulant. The topographical features of powder particles containing 40% milkfat from either anhydrous butteroil or cream are presented (**Figure 3**). The surfaces of powder particles with modified corn starch as the encapsulant were smooth but indented at multiple sites (A & D). Sucrose powder particles were circular in profile and slightly flattened; their surfaces were lumpy or rippled with small semi-circular projections (B & E). Powder particles with all purpose flour as the encapsulant were also slightly flattened and roughly circular in profile with contours of shallow ripples and lumps forming the irregular surfaces (C & F). Surface structures of the powder particles show distinctly different wrinkling and dimpling for different encapsulants. Powder particles

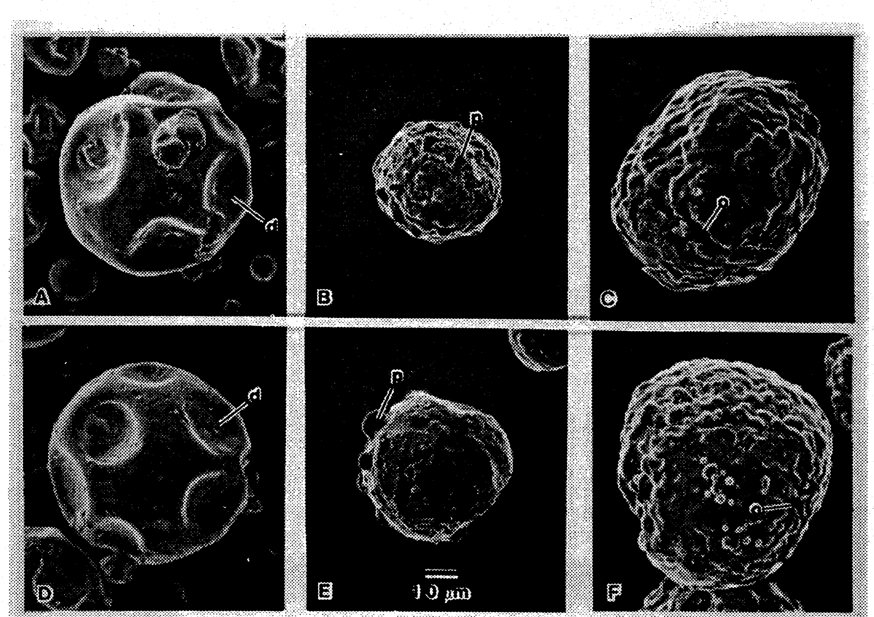

Figure 3.

Scanning electron micrographs of powder particles containing 400 g/kg fat from either anhydrous butteroil or cream. A. Modified corn starch with butteroil showing regular indentations (d), B. Sucrose with butteroil showing superficial protrusions (p), C. All purpose flour with butteroil showing rippled surface and open pores (o), D. Modified corn starch with cream showing indentations (d), E. Sucrose with cream showing protrusions (p) and bar measuring 10 μm, and F. All purpose flour with cream showing rippled surface and open pores (o).

with modified corn starch as the encapsulant showed surface indentations similar to those described for spray dried nonfat milk but without the surface wrinkling (*18*). It has been suggested that dimpling is caused by implosion which occurs during the final stages of drying or by cooling of particles due to rapid water loss from the drying droplet (*19*). Butteroil-containing powder particles with sucrose as encapsulant had a rough-textured surface with many protrusions, whereas particles containing cream as the fat source showed a smoother surface texture with fewer protrusions. Obvious differences in the chemical composition of the encapsulants may further explain differences in the topographical features observed. The chemically modified corn starch, a waxy, low amylose product, was highly acidic (pH 2 to 3). The cross-linking improves resilience. The all-purpose wheat flour had a high protein content; sucrose, a disaccharide, was the least complex of the encapsulants studied.

Modes of Encapsulation

Possible modes of encapsulation of milkfat are schematically illustrated in **Figure 4.** "A" represents one idealized capsule form with a birefringent wall material enveloping milkfat in the core. It is improbable that this idealized structure could be achieved through spray drying. Structure "B" represents real capsules prepared with modified corn starch as the encapsulant. These particles were true capsules, hollow in the middle, with dispersed fine milkfat globules entrapped within the wall matrix. Capsules, prepared with sucrose as the encapsulant, are represented by structure "C"; the particles had a filled core with vesicles (dark) dispersed uniformly throughout the matrix (typical of all disaccharides studied).

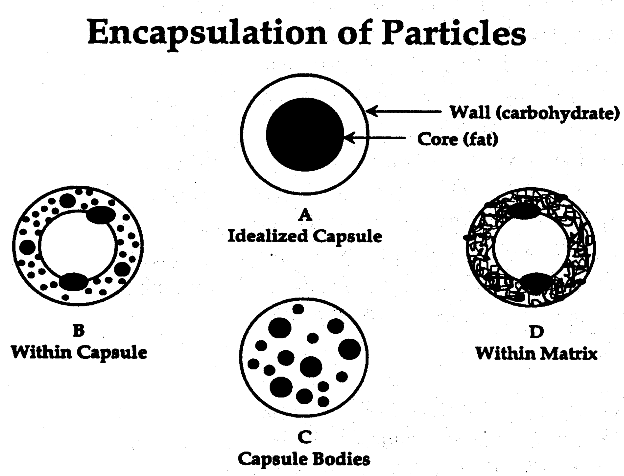

Figure 4.
Illustration of mode of encapsulation of milkfat in various carbohydrate matrices. "A", idealized capsule; "B", Modified corn starch capsule; "C", sucrose capsule, and "D", All purpose flour capsule. Dark spots or bodies represent fat globules.

Powders prepared with all purpose flour as the encapsulant are represented by structure "D"; these particles were also hollow and showed large interior bodies with smaller strands interlaced through the loosely structured particle wall. These particles were not true capsules; consequently, much of the fat content was extractable under our experimental conditions. Modified corn starch or sucrose capsules entrapped the fat within the wall. The inter-spatial fat bodies were not easily accessible to the solvents used. Differential staining of the microparticles with Sudan III and Hematoxylin showed that the birefringent outer capsule contained virtually all of the carbohydrate (blue stain) whereas the core bodies contained the milkfat (red stain) (*20*). Similar structures have been reported for milkfat encapsulated in whey protein matrices (*21*). The milkfat was located in "microcapsules" within the capsules (*20*).

NMR Analysis

To investigate the possibility of complex formation between sucrose and milkfat, as suggested by the appearance of a third high-melting peak in encapsulated sucrose thermograms (*22*), the powders were analyzed by nuclear magnetic resonance (NMR). NMR ^{13}C cross-polarization magic angle spinning nuclear magnetic resonance (CPMAS) spectra were obtained on a Bruker MSL-300 spectrometer (Bruker, USA, Billerica, MA). The high resolution experiments were conducted using magic angle spinning nuclear magnetic resonance (MAS). The spectra were obtained using high powered gated decoupling. The ^{13}C pulse width was 7 μsec pulse with a 5 second recycle time. The sweep width was 25,000 Hz and 2048 data points were collected. 512 transients were collected for each spectrum. A ^{13}C CPMAS spectrum of a powder encapsulated with sucrose and 40% butter oil is presented in **Figure 5**. The CPMAS spectrum shows a low crystalline order sucrose spectrum (110-50 ppm). The high resolution mass spectrum for butter oil in the same powder (Figure 5, mobile phase) shows the presence of the fatty acid side chains (35-18 ppm), a glycerol peak (70-60 ppm), mono-unsaturates (130 ppm) and a carbonyl peak (170 ppm). The ^{13}C spectrum shows no evidence of thermal oxidation of the butter oil caused by the encapsulation process. NMR spectra of sucrose and other polysaccharides have been reported (*23*, *24*). The NMR spectra did not indicate the formation of sucrose-milkfat complexes such as ester linkages under our experimental conditions, suggesting that encapsulation may be stabilized by other interactions such as formation of fat-protein complexes by homogenization before drying (*25*).

Melting Properties

A Perkin-Elmer differential scanning calorimeter, Model DSC-7, equipped with an Intracooler II refrigeration unit was used to measure thermal characteristics (Perkin Elmer Corp., Norwalk, CT). Heating was from -25 to 350°C at 20°C/min after initial cooling to -30°C at 20°C/min. The transition points of the components used for the encapsulation of milkfat are presented in **Table II**. Differential scanning calorimetry profiles showed well-defined melting ranges that were related to encapsulant used. The capsules with sucrose and 40% fat show a disassociation product peak at 239°C; the sample with 60% fat dissociates at a maximum of 236°C. The thermal profiles show two major melting zones for each product containing 40 or 60% encapsulated fat, indicating the melting of the surface or unencapsulated fat and fusion of the wall material. The thermal patterns of these powders are those of true capsules with defined event times for the fusion of the various components comprising the powders. It is known that complexes formed in the presence of saccharides change the melting patterns of the components. Starch degradation studies have shown that in the presence of sugar and emulsifiers, peak temperatures are increased (*26*), suggesting that greater changes are occurring in the starch moiety of this encapsulant as a result of the association with fat. This suggests that it might be possible to tailor capsule rupture temperature by careful choice of encapsulant.

Application in Bakery Products

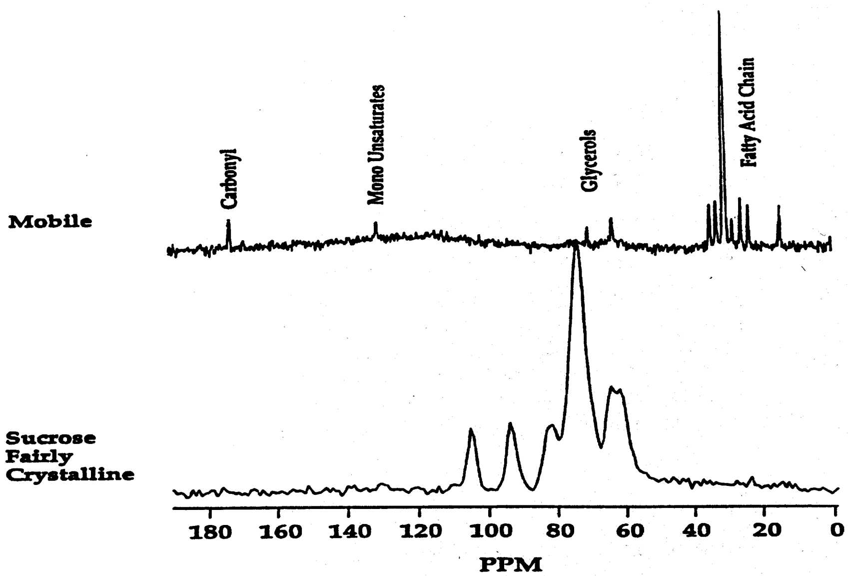

Figure 5.
^{13}C NMR spectrum of powder with 40% butteroil encapsulated in sucrose, showing some crystalline order. Mobile phase shows intact triglyceride structure.

Spray dried powders containing 50% butteroil, encapsulated in sucrose or all-purpose flour, were evaluated by baking biscuits, cookies, or muffins (*27*). Our encapsulated powders were substituted weight-for-weight for shortening in the modified formulations referenced. We selected sugar-snap cookies, biscuits and muffins as products for our baking trials since they contain significant amounts of shortening. When 50% butteroil encapsulated in all purpose flour was substituted for vegetable shortening in the formulations, the weight of flour added as part of the encapsulated butteroil was compensated for by adding an appropriate amount of water (*27*). The substitution of milkfat encapsulated in flour (50% butteroil) or sucrose resulted in increased bulk volume ranging from 3 to 7% over the control formulations made with vegetable shortening.

Table II
Melting Characteristics of Milkfat Encapsulated in Carbohydrate

	Surface fat				Encapsulant load			
	Peak	ΔH	Peak	ΔH	Peak	ΔH	Peak	ΔH
		ΔH = J/g			Peak = °C			
N-starch40	*	*	36.0	10.6	134.4	27.6	--	--
SD			0.0	2.1	2.6	0.4		
N-starch 60	4.0	25.2	39.1	13.6	148.5	19.6	--	--
SD	0.0	0.0	0.1	0.5	0.5	4.3		
M-starch 40	15.2	15.3	34.1	2.5	112.8	58.4	--	--
SD	0.2	0.2	0.1	0.8	0.2	4.1		
M-starch 60	15.8	24.9	36.1	8.3	96.7	9.6	--	--
SD	0.2	4.0	2.1	0.0	0.3	0.8		
Sucrose 40	17.6	14.5	38.0	4.9	183.8	42.5	239.1	23.9
SD	0.6	0.0	0.0	0.1	0.2	0.0	0.1	1.7
Sucrose 60	16.8	24.3	36.2	3.6	177.8	4.7	235.8	15.0
SD	0.2	2.0	0.2	0.2	0.2	2.4	0.2	1.9

*: Insignificant melting peaks. --: No thermal products after carbohydrate peak. N-starch = All-purpose flour and butter oil. M-starch = Modified corn starch and butter oil. 40 and 60 = 40% and 60% milkfat. SD = Standard Deviation [1]Moisture content of powders ranged from 1 to 3%.

Previous work showed that when butter is used as a shortening, it produces a desirable rich flavor and enhances tenderness, especially in yellow cakes. Unfortunately, these qualities may be accompanied by reduced volume which can be offset by the addition of emulsifiers to the formulation (*28*). Baking performance of butter powders in cakes has been reported (*14, 29*). Butter powder could be used successfully as the only source of shortening in cake recipes. Based upon these encouraging reports, we examined the rheological properties, volume, density and porosity of baked products using our encapsulated powders.

Mechanical Testing of Baked Products. Instrumental texture profile analysis (ITPA) was performed on all samples at 25°C with an Instron Universal Testing Machine Model 4201 (Instron, Inc., Canton, Mass). Force-time curves were analyzed. Significant differences were found between the control and the experimental products in cohesiveness, springiness, chewiness and the degree of elasticity (**Table III**). Hardness did not vary significantly, but gumminess and chewiness, which are dependent on product hardness, did vary. With biscuits, the substitution of the butteroil encapsulated in all-purpose flour powder significantly reduced cohesiveness, degree of elasticity, springiness and gumminess; chewiness was slightly, but not significantly, reduced. Both sucrose and all-purpose flour encapsulated powder (50% butteroil) substituted cookies were significantly (P < .05) more cohesive, gummy and springy and much more chewy. These results show that the substitution of the spray dried powder encapsulated in all purpose flour for conventional shortening yielded biscuits and muffins of approximately equal textural quality, while the substitution of the spray dried powder encapsulated in sucrose resulted in muffins and cookies of poorer texture, since more cohesive, elastic and chewy products are desired (*27*).

Table III.
Texture profile analysis[a] of biscuits, cookies or muffins containing encapsulated milkfat as shortening

Sample	Hard(N)	Coh	Gum	Elast	Sprgn	Chew(mm)
			Biscuits			
Control	3.34^d	0.39^d	1.28^e	0.40^d	6.00^d	7.67^d
FLB050[b]	3.09^d	0.36^e	1.10^d	0.33^e	5.14^e	5.55^d
			Cookies			
Control	194^d	0.24^f	45.4^e	0.40^f	1.26^f	57.1^e
SUB050[c]	192^d	0.33^d	61.6^d	0.49^d	1.58^d	96.2^d
FLB050	242^d	0.30^e	72.1^d	0.45^e	1.34^e	96.0^d
			Muffins			
Control	10.6^d	0.29^d	3.06^d	0.54^d	8.93^d	30.0^d
SUB050	10.3^d	0.27^e	2.81^d	0.42^f	7.92^e	22.2^e
FLB050	10.1^d	0.30^d	3.04^d	0.49^e	8.77^d	26.7^f

[a] Hard: Hardness, height of first peak; Coh: Cohesiveness, ratio of area under second peak to area of first peak (A_2/A_1); Gum: Gumminess, product of Hard x Coh x 100; Sprgn: Springiness; Elast: Degree of elasticity; Chew: Chewiness, product of Hard x Coh x Sprgn.
[b] FLBO50: 50% butteroil encapsulated in all purpose flour.
[c] SUBO50: 50% butteroil encapsulated in sucrose.
Different superscripts in same column for each product significantly different by Analysis of Variance at P < .05

Summary
Milkfat globules were successfully encapsulated when sucrose was used as the encapsulating agent; no voids or large vacuoles were present. With minimal extractable fat, powders with sucrose were structurally stable, suggesting good milkfat retention. Spray dried encapsulated milkfat powder shows potential for use as a food ingredient in such products as dry bakery mixes. Well-defined melting ranges are identifiable in DSC profiles; melting temperatures for capsule rupture and release of the milkfat content depend on type of encapsulating agent chosen. Low levels of extractable fat show protection of the encapsulated butteroil, when sucrose is the encapsulant, which is essential in maintaining milkfat quality. We have demonstrated that milkfat encapsulated in functional encapsulating agents may be substituted for traditional shortening in three types of baked products with considerable success. Advantages of microencapsulation include greater ease of shipping and handling, possibilities for compression to save packaging and storage space and possible protection against oxidative deterioration at ambient temperatures.

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