

Flow and Compaction of Spray-Dried Powders of Anhydrous Butteroil and High Melting Milkfat Encapsulated In Disaccharides

C.I. ONWULATA, P.W. SMITH, and V.H. HOLSINGER

ABSTRACT

Anhydrous butteroil or a high-melting milkfat fraction prepared from it were encapsulated in lactose, maltose or sucrose matrices by spray drying to compare functional characteristics. Both types of milkfat were retained efficiently (>90%) within the encapsulants at 40% fat level. Retention declined as fat concentration increased from 40 to 60%, regardless of melting temperature of the milkfat. Encapsulant or type of milkfat did not alter bulk properties. Powders withstood compaction pressures <60% with minimal capsule rupture. Powder functionality may be modified by varying disaccharide choice and milkfat melting properties.

Key Words: anhydrous butteroil, spray drying, milkfat, disaccharides, encapsulation

INTRODUCTION

MICROENCAPSULATION TECHNIQUES have been used to manufacture free flowing powders containing milkfat (Onwulata et al., 1994a; Young et al., 1993). The milkfat is entrapped within a matrix formed by protein and carbohydrates that may protect the fat from oxidative deterioration during storage if fat-containing microcavities are successfully formed during the drying process (Imagi et al., 1992). The formation of microcapsules is material dependent; for example, oligosaccharides may form multi-component microchambers within a capsule (Lichtenthal et al., 1992). Formation of microcapsules during spray drying improved physical properties and retained high levels of milkfat (Onwulata et al., 1994a).

Milkfat is composed of triglycerides ranging from volatile low melting to high melting (stearine) fractions. Fractionation of milkfat through various separation processes is based on differential crystallization of triglycerides at different temperatures. The high melting fractions may be used as cocoa butter substitutes or as shortening for specialty baked goods such as Danish pastry (Barts, 1991). The low melting fractions with a concentration of flavors, vitamins and pigments could be used for production of a soft butter, spreadable at refrigerator temperatures, or for formulation of parenteral products. Microencapsulation of milkfat fractions in functional matrices provides potential for new uses in food formulations (Boudreau and Arul, 1991; Timms, 1991). For example, the common disaccharides lactose, maltose and sucrose have varying degrees of solubility and sweetness, suggesting that milkfat encapsulated in such matrices might have utility in confectionary products (Campbell and Pavlasek, 1987).

Disaccharides have been used as encapsulating agents. Volatiles have been entrapped and retained in sucrose, maltose or lactose through conversion from amorphous to crystalline state by freeze drying (Flink and Karel, 1970). Maltose retained volatiles within micro-regions through molecular associations (Chirife and Karel 1974). Volatile retention is high in such powders through the formation of impermeable surface membranes, increased resistance to diffusion at low water contents, and the

formation of inclusion complexes (Flink and Karel, 1970; Menting and Hoogstad, 1967).

Flow rate of powders containing milkfat may depend on properties of the wall materials used for encapsulation (Peleg, 1978) and is retarded by the presence of unencapsulated fat on powder particle surfaces (Onwulata et al., 1994b). Moisture sorption properties and crystallization temperatures of the disaccharides influence the transition from amorphous to crystalline state and are related to powder stickiness and caking. Crystallization is important when encapsulated milkfat is stored without special packaging, as crystallization in the presence of high relative humidity causes capsule rupture, exposing the milkfat to air (Onwulata and Holsinger, 1995).

Our objective was to investigate the physical-chemical properties and structures of powders containing anhydrous butteroil or a high melting oil fraction made from it, with disaccharides of varying solubility and sweetness used as encapsulating agents. We determined some physical characteristics of such powders; properties such as bulk density, butteroil retention, powder flowability, particle size distribution, cohesiveness, compressibility and compaction were evaluated.

MATERIALS & METHODS

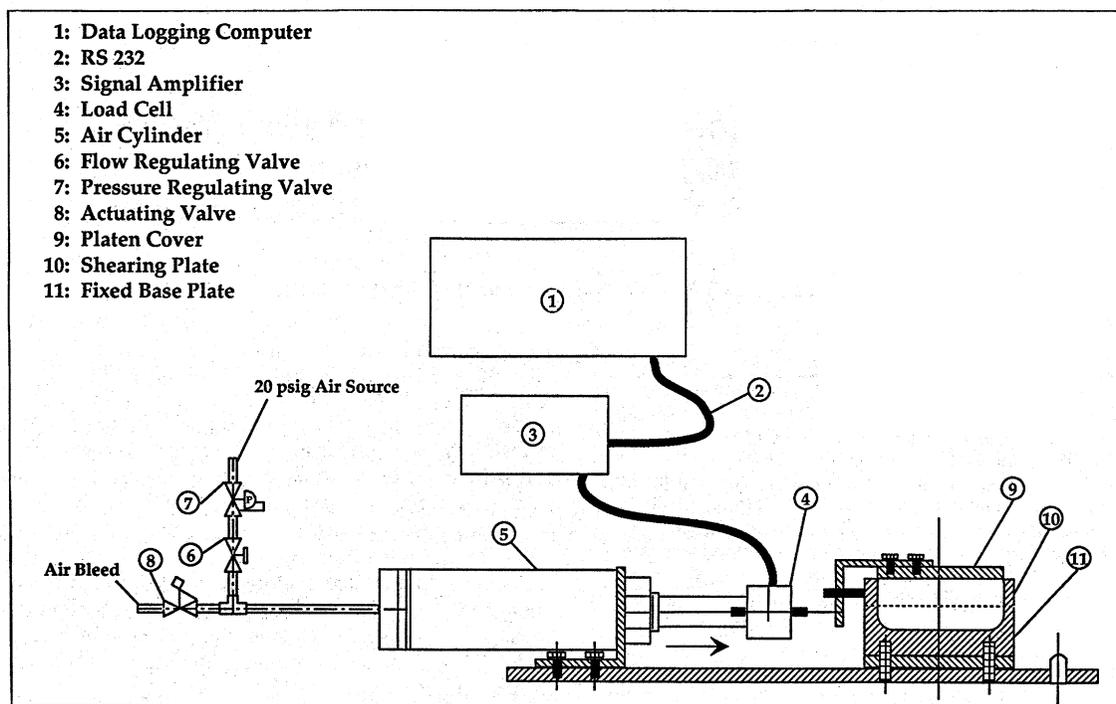
ANHYDROUS BUTTEROIL (BO) was purchased from a commercial manufacturer (Land-O'-Lakes, Inc., Minneapolis, MN). A high melting (stearine) fraction (HSBO) was made by slow crystallization and separation of the fractions. BO was heated to 60°C, cooled quickly to 10°C, then gradually warmed and held at 35°C for 72 hr. The BO was continuously stirred (60 rpm), allowing for the HSBO to solidify and float. HSBO crystals were removed by frequent skimming from the top of the separator. Peak melting point of the HSBO fraction was 42°C by differential scanning calorimetry.

The disaccharides used were sucrose (Domino's, Domino Sugar Corp., New York, NY), lactose (Swiss Valley Farms Co., Davenport, IA), and maltose (Penta Manufacturing, Livingston, NJ). The emulsifying agent was composed of mono- and diglycerides (American Ingredients Co., Kansas City, MO). Sodium caseinate (New Zealand Milk Products, Santa Rosa, CA) served as protein source.

Sample preparation was carried out by following a full $2 \times 2 \times 3$ factorial design, that was completely randomized and replicated two times. Encapsulated powders were formulated to have 40 or 60% BO or HSBO, 5% emulsifier, 4.5% sodium caseinate and the remainder, disaccharide. Either anhydrous butteroil (BO) or HSBO was warmed to 60°C, and blended with the emulsifying agent. The encapsulant was dry blended with the sodium caseinate and dispersed in water to form a slurry of about 25% total solids. The mixtures were combined (40% total solids), heated to 63°C with constant stirring, and homogenized at 17.2 MPa with a Manton-Gaulin Triplex homogenizer (Model 100 DJF3 855X, APV Gaulin, Inc., Everett, MA). The homogenized emulsion was spray-dried in a compact dryer (APV Crepaco, Inc., Attleboro Falls, MA) at inlet temperature 193–196°C and outlet temperature 82–89°C. The slurry was passed through a centrifugal atomizer at 25,000 rpm. Powders were produced batchwise; residual powder was swept from the dryer wall every 30 min. Powders were stored at 4°C until used.

Moisture was determined in triplicate by AOAC (1990) method #1927.05 by drying under vacuum for 4 hr at 102°C. Water activity of powders was measured in duplicate at 26°C with a water activity meter (Decagon Devices, Inc., Pullman, WA).

Extractable fat was determined in duplicate by dispersing 10 g of powder in 50 mL of carbon tetrachloride (Warning: carbon tetrachloride



Cohesion Testing Device

Fig. 1—Device for measuring powder cohesion.

is a known carcinogen and care must be taken with its use) and shaking for 15 min with an orbital shaker (Labline Instruments, Melrose Park, IL) at 200 rpm (Anonymous, 1978). The soluble fraction was decanted, filtered and the solvent evaporated, leaving the extracted fat. Extractable fat was expressed as the amount of fat recovered from the powder, divided by 10. The percent fat retained (ϵ) was determined as follows:

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

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

Bulk densities of all powders were determined by dividing the weight of powder (g) filling a 200-mL stainless steel cylinder (Anonymous, 1978) without tapping, by the cylinder volume in cm^3 . Six density measurements (g/cm^3) were made for each treatment.

Particle size distributions were calculated from sets of optical photomicrographs of powders sprinkled on one-cm-long segments of double stick adhesive tape (3M Company, St. Paul, MN) 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 from each treatment 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.

Flow characteristics were evaluated in triplicate by letting powder flow through a funnel to form a heap; angle of repose (θ), a measure of the flowability of a powder, was calculated from the base angle formed by the heap of powder as $\theta = \tan^{-1} h/r$; where "h" is the height of powder, and "r" is the radius (Sjollem, 1963). The relative flow of the powder with time was measured by permitting 80 g to flow through metal funnels of outlet diameter 1.27 cm with gentle shaking (FMC/Synthron, Homer, PA). Time of flow was recorded. Relative flow rate was calculated as powder weight divided by time (g/sec).

Cohesiveness of encapsulated powders was determined by first compressing 25g of powder in a 64 cm^3 cylinder under a 220-g load. The 220-g load was released and was replaced by a 175-g platen cover to hold the compressed powder in place. The compressed powder com-

pletely filled the cylinder (Fig. 1). The force required to push the shearing plate (10) through the compressed powder was measured by the load cell (4); the peak force recorded is the cohesive force (Mohnesin 1970). The greater the force needed, the more cohesive the powder. The ramming piston was air-driven (5) with the force transducer interfaced to a PC for data-logging and graphics display.

Compressibility was determined by compressing the powders from 10 to 40% of the original volume with an Instron Universal Testing machine (Model 4200, Instron Corp., Canton, MA). Compression was at a cross-head speed of 10 cm/min . The cells used were 30 mm h \times 45 mm diam. Compressive pressure (g/cm^2) measurements were repeated four times for each treatment (Moreyra and Peleg, 1980).

Powder compaction was done to test the mechanical stability and block forming characteristics of each powder. A sample (150 g) of encapsulated powder was compacted in a brass vessel of 60 mm depth and 90 mm dia. A hydraulic laboratory press (Carver Laboratory, Menomonee, WI) with fixed piston was used for compaction. Maximum load at a pre-set compaction level was recorded in the range 45 to 75% compaction.

Results were analyzed for trends with the General Linear Models Procedure of the Statistical Analysis System (SAS Institute, Inc., 1991). Mean value separations were determined by Duncan's Multiple Range test and significance of differences was established at $p \leq 0.05$.

RESULTS & DISCUSSION

MICROENCAPSULATION by spray drying of fats is usually done to enhance handling properties, especially when blending with nonfat ingredients. Such powders must resist capsule rupture during packaging, shipping and storage since desirable flow characteristics are impaired by unencapsulated fat on particle surfaces. We previously demonstrated that free-flowing powders retaining >95% of fat within the capsules could be successfully spray dried with sucrose as encapsulant and anhydrous butteroil as fat source (Onwulata et al., 1994a).

We compared the physical properties of spray-dried powders with BO or HSBO encapsulated at two levels in lactose (Table 1), maltose (Table 2) or sucrose, (Table 3). Moisture content ranged from 0.65 to 2.52% and depended on the sugar used for

Table 1—Physical properties of butteroil (BO) or a stearine fraction (HSBO) made from it, encapsulated in a lactose matrix

Product	Moisture %	A_w	Fat extracted %	ρ g/cm ³	Flow g/sec	θ degrees	ϵ %	Cohesion kg/cm ²
LBO40	1.65 ^a	0.25 ^a	2.75 ^b	0.26 ^a	16.7 ^a	44.3 ^a	93.1 ^a	30.0 ^a
SD ^d	0.40	0.03	1.06	0.01	0.8	0.0	1.9	6.4
LBO60	1.32 ^a	0.28 ^a	13.6 ^a	0.23 ^b	18.3 ^a	43.3 ^a	77.3 ^b	32.6 ^a
SD	0.13	0.02	0.5	0.01	1.9	0.2	0.6	0.9
LHSBO40	1.74 ^a	0.30 ^a	3.50 ^b	0.25 ^a	16.9 ^a	45.7 ^a	91.2 ^a	30.3 ^a
SD	0.28	0.08	0.71	0.00	0.0	5.2	1.2	7.8
LHSBO60	1.72 ^a	0.32 ^a	17.0 ^a	0.25 ^a	15.0 ^a	45.5 ^a	71.7 ^c	28.7 ^b
SD	0.02	0.04	2.8	0.01	2.2	2.1	3.3	5.5

^{a-c} Values in the same column followed by different letters are significantly different at $p < 0.05$.

^d SD = Standard Deviation.

Table 2—Physical properties of butteroil (BO) or a stearine fraction (HSBO) made from it encapsulated in a maltose matrix

Product	Moisture %	A_w	Fat extracted %	ϵ %	ρ g/cm ³	Flow g/sec	θ degrees	Cohesion kg/cm ²
MBO40	2.40 ^a	0.26 ^a	2.25 ^c	94.4 ^a	0.26 ^a	14.8 ^a	46.2 ^a	14.8 ^c
SD ^d	0.07	0.03	1.06	1.9	0.01	1.2	3.8	7.6
MBO60	2.12 ^a	0.32 ^a	8.62 ^b	85.6 ^a	0.23 ^b	14.2 ^a	42.9 ^a	24.1 ^a
SD	0.01	0.02	1.94	2.3	0.04	2.6	3.1	2.7
MHSBO40	2.30 ^a	0.32 ^a	2.87 ^c	92.8 ^a	0.25 ^a	15.8 ^a	44.5 ^a	16.9 ^b
SD	0.03	0.10	0.18	0.3	0.01	0.7	1.0	1.8
MHSBO60	2.52 ^a	0.28 ^a	11.5 ^a	80.8 ^a	0.24 ^a	17.2 ^a	43.1 ^a	19.9 ^b
SD	0.10	0.02	3.5	4.2	0.03	3.4	1.3	0.5

^{a-c} Values in the same column followed by different letters are significantly different at $p < 0.05$.

^d SD = Standard Deviation.

Table 3—Physical properties of butteroil (BO) and a stearine fraction (HSBO) made from it encapsulated in a sucrose matrix

Product	Moisture %	A_w	Fat extracted %	ϵ %	ρ g/cm ³	Flow g/sec	θ degrees	Cohesion kg/cm ²
SBO40	0.85 ^a	0.21 ^b	1.86 ^c	95.3 ^a	0.29 ^a	19.2 ^a	42.7 ^a	35.8 ^a
SD ^d	0.02	0.01	0.50	2.8	0.00	4.9	0.4	6.4
SBO60	0.67 ^a	0.24 ^a	4.00 ^b	93.3 ^a	0.24 ^b	14.6 ^a	43.6 ^a	30.0 ^b
SD	0.03	0.05	0.71	0.8	0.02	3.6	3.8	5.5
SHSBO40	0.87 ^a	0.19 ^b	3.00 ^b	92.5 ^a	0.25 ^a	16.9 ^a	43.6 ^a	29.3 ^b
SD	0.22	0.01	0.88	3.8	0.03	4.5	0.7	5.5
SHSBO60	0.65 ^a	0.26 ^a	5.50 ^a	90.8 ^a	0.24 ^b	16.4 ^a	41.9 ^a	31.6 ^b
SD	0.03	0.04	0.88	2.5	0.03	4.0	3.7	0.5

^{a-c} Values in the same column followed by different letters are significantly different at $p < 0.05$.

^d SD = Standard Deviation.

encapsulation; lowest moistures were found in powders encapsulated in sucrose. Although the disaccharide encapsulants are in the (mostly) amorphous state after drying, the higher moistures in the maltose- and lactose-containing powders may indicate formation of some crystal structure since both sugars have a mono-hydrate crystalline structure. Moisture content tended to be lower in powders with 60% fat with exception of HSBO encapsulated in maltose and also varied ($p < 0.05$) with type of fat, BO or HSBO. Moisture content is important in milkfat retention in the microcapsules. As moisture content increases above a critical level, crystallization of sugar occurs, the encapsulating structure weakens and milkfat extraction increases (Onwulata and Holsinger, 1995).

Water activity of the disaccharide capsules varied ($p < 0.05$) from 0.19 to 0.32; lowest water activities were found with powders encapsulated in sucrose. Many physical properties (e.g., compression, cohesion, compaction, and bulk properties) are influenced by water activity. Changes in water activity following crystallization influence agglomeration and caking in stored powders. Peleg and Mannheim (1977) reported that caking did not occur at ambient temperature at water activity < 0.4 . Low moisture content and water activity in encapsulated food powders contribute to improved physical and bulk properties.

The amount of extractable fat varied depending on encapsulant and level of fat. Most fat was extracted from lactose powders with 60% BO or HSBO. More fat ($p < 0.05$) was extracted from powders from HSBO in all cases. In terms of fat retention, sucrose was the most efficient encapsulant (91 to 95% fat retained). Retention declined with maltose powders (81 to 94%)

and was least efficient with lactose (72 to 93%). Retention declined sharply as fat level increased from 40 to 60%, with an average decline for lactose of 17%, for maltose, 10%, and for sucrose, 2%. Variation of percent fat retained after encapsulation has been related to the disaccharide group as in comparison with other encapsulating carbohydrates (Onwulata et al., 1994a). Fatty acids are reported to be tightly bound to proteins of the milkfat globule membrane by covalent linkages (Keenan and Heid, 1982). Certain triglycerides, in binary systems, form new stable polymorphs in a compound order (Engstrom, 1992). Strongly polar interactions between milk proteins and milk lipids through hydrogen bonds and electrostatic attraction have been reported during emulsion formation (Aynie et al., 1992). The potential is known for disaccharides to form backbones of complexes (Lichtenthal et al., 1992). Also milkfat-protein complexes form (Henstra and Schmidt, 1970; Fox et al., 1960) in a high shear (17.2 MPa), high temperature (199°C) environment. Thus complexes that are not easily extracted from small vesicles within the capsules may be formed. We suggest that in the presence of good emulsifiers such as sodium caseinate and mono- and di-glycerides, sucrose, followed by maltose or lactose, may form stable protein-lipid-saccharide complexes. The complexes were resistant to lipid extraction under our experimental conditions. Other benefits of complex formation may be an increase in antioxidant activity by casein in casein-sugar mixtures, and the presence of Maillard reaction products which can enhance flavor (McGookin and Augustin, 1991).

The product density range was small for all powders (0.23–0.29 g/cm³). Product density decreased slightly at higher milkfat

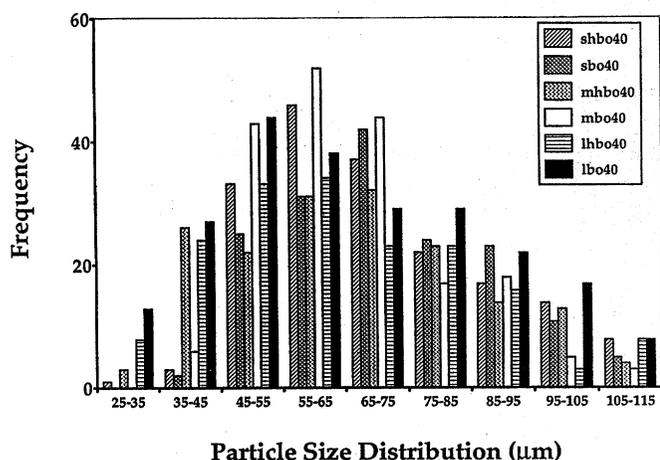


Fig. 2—Particle size distribution of anhydrous butteroil or a high melting (stearine) fraction of milkfat encapsulated in lactose, maltose or sucrose matrices.

concentrations. This was because less wall material was used for the 60% formulation, as the disaccharides were denser than fat.

The particle size distributions of the encapsulated powders (Fig. 2) were similar with a distribution range of 25 to 115 µm; the most frequent size was around 45 to 85 µm. Powder bulk properties have been related directly to powder composition, particle sizes, and moisture content (Moreyra and Peleg, 1981; Peleg, 1983). As mentioned, for encapsulated powders, surface fat and increased hygroscopicity of the encapsulant affect handling, storage and flow characteristics adversely (Onwulata and Holsinger, 1995; Onwulata, et al., 1994b). Product density and particle size affect space filling in bulk packages. Coarse particles occupy more space and fine particles tend to be more dense (Conovas et al., 1987).

The flow properties were compared for the powders encapsulated in lactose (Table 1) maltose (Table 2) or sucrose (Table 3). Timed flow rate through a funnel ranged from 14.2 to 19.2 g/sec. No significant differences occurred in powder flow attributable to type of milkfat or fat level. As a group, powders encapsulated in maltose tended to flow more slowly possibly because of their higher moisture levels. Sucrose powders had the highest flow rates followed in order by lactose and maltose.

Angles of repose for powders encapsulated in lactose, maltose or sucrose were also compared. They were not different ($p > 0.5$) for any of the powders. The type of milkfat made no difference in flowability by this measure as the free-flow angles ranged from 42 to 46 degrees. Powder flow is normally influenced by such factors as amount of surface fat, hygroscopicity of encapsulant, particle size and other bulk properties. Some correlations have shown flow properties were proportional to bulk density (Moreyra and Peleg, 1981). Uniformity in particle size and shape improves powder flow (White et al., 1967). Our encapsulated particles fell within a relatively narrow particle size range, with relatively uniform distribution and shape. Powders with sucrose as encapsulant contained little extractable fat, especially at the lower fat levels. Previous work (Onwulata et al., 1994b) with milkfat encapsulated in sucrose showed little or no milkfat present on the particle surface to interfere with flow. Compared to published data for other food powders, powders of milkfat encapsulated in disaccharide matrices were free flowing (Sjollema, 1963).

Inter-particle cohesion measurements showed that maltose powders were less cohesive than those from lactose and sucrose. Cohesion tended to increase with increasing fat level and type of fat appeared to have no effect on cohesion. Cohesive powders have been proposed to be those where inter-particle forces cause "bridging;" the mechanism for "liquid bridging" includes such

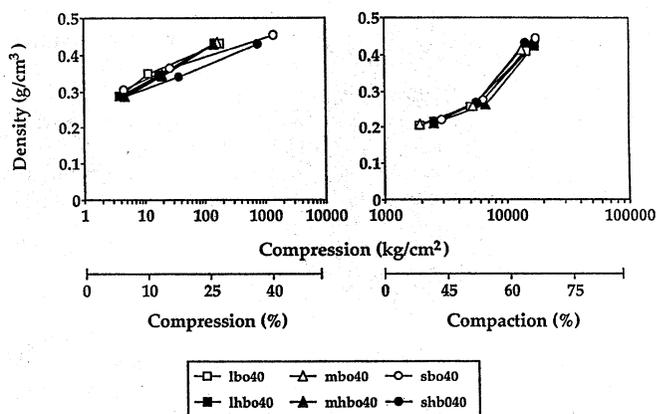


Fig. 3—Compression and compaction of spray-dried powders with anhydrous butteroil or a high melting (stearine) fraction of milkfat encapsulated in lactose, maltose or sucrose matrices at 40% fat level.

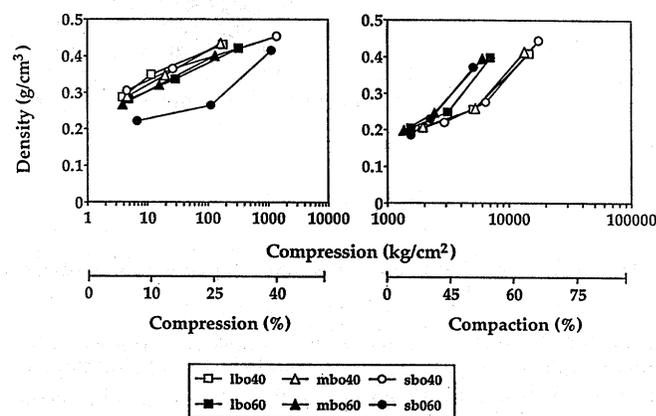


Fig. 4—Compression (Instron) and compaction (hydraulic press) of spray-dried powders with anhydrous butteroil encapsulated in a lactose, maltose or sucrose matrix at 40 or 60% fat level.

factors as hygroscopicity of materials, moisture content, and wettability (Peleg, 1977). Such factors suggest that encapsulated high fat powders would lump and cake, but we observed no lumpiness. The powders remained very free flowing as long as they were protected from high ambient moisture (Peleg et al., 1973; Makower and Dye, 1956).

Compression and compaction of encapsulated powders with 40% fat (Fig. 3) showed that bulk properties were not different ($p > 0.5$). Powders with the same amount of fat (40%) did not differ in force needed for compaction regardless of differences in milkfat fractions. Differences were established when the amount of encapsulated fat increased from 40 to 60% (Fig. 4) where all powders with 60% butteroil required less force to compact. The powder with 60% butteroil encapsulated in sucrose was less compressible than were the other powders. Non-linear compression patterns were observed for encapsulated powders, similar to those reported for nonfat dry milk (Hanrahan and Kontson, 1965). Though Moreyra and Peleg (1981) reported deformation patterns that were different, their work was with dry non-cohesive powders. Stickiness, a property of amorphous food powders in conjunction with natural viscous forces at or near the "sticky point temperature" of the powders, would be expected to improve both compaction and compression (Wallack and King, 1988; Makower and Dye, 1956). Powders encapsulated in sucrose, with the least amount of moisture, least extractable fat and lowest water activity, compressed less when lower force was applied (Fig. 3 and 4) and were most cohesive (Table 3). Nondestructive powder compaction was observed up

to 60% compression by volume of encapsulated powder when the hydraulic press was used. Compression >65% ruptured the micro-capsules and released the milkfat. Powder compaction reduces storage space. Webb and Hufnagel (1943) reported a 42% saving in space with compressed dry whole milk. Though differences in space saved would depend heavily on type of powder, we could compress highly hygroscopic powders (Onwulata and Holsinger, 1995) up to 60% without capsule rupture or release of milkfat.

CONCLUSIONS

BO, ENCAPSULATED IN LACTOSE, maltose or sucrose matrices by spray drying provided relatively free flowing powders. No problems were encountered in encapsulation of a high melting milkfat fraction (HSBO) which could provide a specialty shortening for baked goods. Powders made with sucrose as encapsulant had best physical properties in terms of flow rate and percent fat retained; they were least compressible but powders with maltose and lactose were equally compressible. Powders with sucrose are useful ingredients in sweet foods while those with lactose could provide browning capability while contributing minimum sweetness. 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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