

# Properties of Single- and Double-Encapsulated Butteroil Powders

## ABSTRACT

Spray-dried powders with 50% butteroil encapsulated in sucrose were double-encapsulated by dispersion in a molten matrix of vegetable waxes (high-pressure-treated by a patented process) followed by pressure treating at 414 KPa and sieving. A 20–40% increase in particle size resulted from double encapsulation and powders were less flowable ( $P < 0.01$ ). Scanning Electron Microscopy of double-encapsulated powders showed one or more sucrose-encapsulated particles embedded in a matrix of solid vegetable wax. Double coating reduced moisture uptake by 20%, possibly ameliorating the need for special packaging during storage. The flow and mechanical behavior of the encapsulated powders were different ( $P < 0.05$ ) from the other powders studied.

**Key Words:** butteroil, milkfat, encapsulation, double coating

## INTRODUCTION

ENCAPSULATION OF POWDERS WITH DIFFERENT components is expected to become increasingly important in foods especially for substitution of bioactive additives (Jackson and Lee, 1991; Duxbury and Swientek, 1992). Compound powders with "soft" cores provide means for protecting sensitive components with pharmaceutical benefits. Protecting loss of active nutrients, such as carotenoid activity during dehydration, could be minimized by encapsulation of beta-carotene (Wagner and Warthesen, 1995). Marine omega-3 fatty acids have been encapsulated in a starch/oil emulsion and spray-dried to provide for this protection in food powders (Andersen, 1995).

The entrapment of sensitive ingredients within a continuous film or coating can protect them from environmental factors such as moisture, air or light. Encapsulation, however, leads to demonstrable difficulties in handling, due to changes in bulk properties of the powders. This was particularly true for encapsulated butteroil, where the encapsulated powders showed a propensity for stickiness and lumping (Onwulata et al., 1995, 1996; Konstance et al., 1995).

The amount of fat retained after encapsulation of butteroil is dependent on the carbohydrate used as the encapsulating agent (Onwulata et al., 1994). The main purpose of preparing spray-dried fats is to enhance their handling properties, and stability during storage, transport and blending with nonfat ingredients. Butteroil, to be made into a powder, requires a carrier because it contains appreciable amounts of low melting triglycerides. As the fat content of the capsule increases, the choice of carrier constituents becomes

more and more important, as fats on the surface of powders retard flow (Onwulata et al., 1995).

Several disaccharides have been used as encapsulating agents. Volatiles have been entrapped and retained in sucrose, maltose or lactose by freeze-drying the amorphous sugar to the crystalline state (Flink and Karel, 1970). There has been remarkably high volatile retention in such powders. The surface membranes are impermeable with high resistance to diffusion at low water content or to the formation of inclusion complexes (Flink and Karel, 1970; Menting and Hoogstad, 1967). A second encapsulating coat may provide a better moisture barrier. Such a barrier could be created through a combination of waxes coating the surfaces to form the double-coat. Minimizing moisture uptake is extremely important for encapsulated powder stability and effectiveness (Onwulata et al., 1995).

Proper choice of material for the second coat would enhance delivery of the functional content (Pothakamury and Barbosa-Cánovas, 1995). This is essential for controlled release of components in foods, an aspect that is becoming increasingly useful for functional powders. Our objective was to investigate the physical properties and structures of double-encapsulated powders containing anhydrous butteroil compared to characteristics of single-encapsulated powders. We compared some physical characteristics of the powders and properties such as bulk density, powder flowability, cohesiveness, and compressibility.

## MATERIALS & METHODS

BUTEROIL WAS OBTAINED FROM A COMMERCIAL manufacturer (Land O'Lakes, Inc., Arden Hills, MN). The encapsulating agent was granulated sucrose. Spray-dried powders containing 50% butteroil were prepared in our pilot plant as previously described (Onwulata et al., 1994). Encapsulated powders

were formulated to have 50% butteroil, 5% emulsifier, and 5% skim milk powder, with the remainder encapsulant. The emulsifiers used were mono and di-acylglycerides (American Ingredients Co., Kansas City, KS). The processing sequence for the single coat capsule was as follows: Sucrose was dry-blended with nonfat dry milk solids and the mixture dispersed in water to form a pasty slurry of 25% total solids. The anhydrous butteroil and the emulsifier were heated to 45°C with stirring. The two blends were combined (40% total solids) and mixed for 5 min with a milk stirrer, after which the slurry temperature was slowly raised to 62.8°C with constant stirring. The mixture was 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) at an inlet temperature of 193.3–196.1°C and an outlet temperature of 82.2–87.8°C. Powders were produced batch-wise, collected from the dryer after 30 min and stored at 4°C.

Double encapsulation was done with the application of a mixture of vegetable waxes (hydrogenated stearines), Sterotex NF® (Abitec Corp., Columbus, OH) and food grade linear alcohol polymers, Unilin 350® (Petrolite, Tulsa, OK) at a 99:1; Sterotex to Unilin ratio. The waxes were melted at 105°C. The molten waxes which are normally in the alpha-form were converted to the more stable beta form using the Beta® process device (Encapsulation Systems Inc., Sharon Hills, PA). The molten wax was sprayed onto the preformed single-encapsulated particles. The moist particles were subjected to another pressure shock wave at 414 KPa, to cause the wax to form a second coat on the particles. The waxed powders were subsequently cooled, sifted through a 20-mesh sieve and stored. The process and the M-CAP® device used for double-encapsulation have been reported (Redding 1990; 1993).

The single-encapsulated powders passed through a 500- $\mu$ m sieve, and the double-encapsulated powders passed through a 1000- $\mu$ m sieve.

## Analyses

The moisture contents of the powders were determined by drying in a vacuum oven at 102°C for 4h (AOAC, 1984). Moisture determinations were made in triplicate.

Moisture uptake by the powders was determined at 25°C by equilibrating 10-g pow-

der samples at 52% relative humidity. Vapor pressure was maintained with a saturated salt solution of  $Mg(NO_3)_2$  (Rockland and Nishi, 1980). Measurements were made in duplicate.

The mass flow of the powder (g/s) was measured by permitting 80g to flow through funnels of outlet dia 30 mm with gentle shaking using a Syntron Shaker (FMC/Syntron, Homer, PA) at the setting of 40, for those powders that would not flow without mechanical agitation (double-encapsulated powders). Time of flow was recorded and relative flow rate was calculated as powder weight (g) divided by time (s). Flow rates were measured in triplicate.

Particle density was determined in triplicate with an air pycnometer (Horiba Instruments Inc., Irvine, CA).

Bulk densities of all powders were determined by dividing the powder mass (g) contained in a 200-mL stainless steel cylinder (A/S Niro Atomizer, Copenhagen, Denmark) by its volume ( $cm^3$ ). Packed bulk densities were calculated from the weight of powder contained in the cylinder after being tapped 100 times. Density measurements ( $g/cm^3$ ) were done in triplicate.

Compressibility was determined as described by Moreyra and Peleg (1980). The powders were carefully poured into a sample cell (30 mm high/45 mm dia) and the loose density ( $g/cm^3$ ) was determined from the weight and known volume. The sample cell was mounted on the base plate of a Model 4200 INSTRON Universal Testing Machine (Instron, Canton, MA). The powders were compressed at a crosshead speed of 10 mm/min using a 50-kg load cell to a preselected force of 40 kg. Powder compressibility was determined by evaluating the slope of the plot of bulk density and the logarithm of compressive stress ( $1 < \log < 4$ ) using  $D = a + b \log$ ; (Sone, 1972); where  $D$  = bulk density ( $g/cm^3$ ) at corresponding  $\sigma$  = compressive stress ( $g/cm^2$ ) and  $a$ ,  $b$  = empirical constants with "b" representing compressibility. Three data points were used.

Particle sizes were determined optically with an Accusizer® 770 (Particle Sizing Systems, Inc., Santa Barbara, CA). The particles were counted individually with a dry powder attachment and the population distribution was calculated from accumulated counts.

The topography of intact and fractured particles was examined by secondary electron imaging using a scanning electron microscope (Model 840A, JEOL, USA, Peabody, MA). Three methods of preparation were used. For superficial structure of intact particles, small samples of powder ( $< 1$  cc) were applied to an adhesive layer (Spot-O-Glue, Avery, AZ) affixed to 12-mm-dia A1 stubs. Excess particles were removed by directing a jet of dry nitrogen gas (at 276 KPa) at the surface of the stub. For superficial structure of dehydrated particles, small samples of powder were immersed in ice-cold ethanol, followed by critical point-drying

from liquid carbon dioxide. The dried particles were applied to A1 stubs as described above. For topographical study of internal particle structure, single selected powder particles were immersed in liquid nitrogen and manually cut with the edge of a cold stainless steel razor blade, or broken with cold tips of Dumont #5 tweezers (Electron Microscopy Sciences, Ft. Washington, PA). After 'fracturing', the pieces of particles were placed in an A1 foil envelope under liquid nitrogen and transferred to a freeze-drying apparatus. Dried pieces of particles were glued to A1 stubs with internal faces oriented upwards. All samples were coated with a thin layer of Au in a sputter coater (Model LVC-76, Plasma Sciences, Inc., Lorton, VA).

Extractable fat was determined by dispersing 10g powder in 50 mL petroleum ether and shaking for 15 min (Anonymous, 1978). The soluble fraction was filtered and the solvent was evaporated, leaving the fat and wax. Samples were analyzed in triplicate. Extractable fat (weight fraction) was expressed as the weight of fat recovered from the powder, divided by the weight of original powder. The weight fraction of extractable residual fat was inversely related to the quality of encapsulation. That is, the lower the extractable fat, the more effective was the encapsulation.

## RESULTS & DISCUSSION

MEASURED PHYSICAL PROPERTIES OF SINGLE- and double-encapsulated powders were compared (Table 1). The moisture content was characteristic of powders encapsulated with sucrose (Onwulata et al., 1995). Double-encapsulated powders had higher moisture, 36% more than single-encapsulated powders. The moisture content of the particles was below the critical water activity ( $0.4 a_w$ ) level and did not lead to crystallization of sucrose which would have been detrimental to flow properties.

The densities, both loose and bulk, were typical of encapsulated powders, and were lower than those of the sucrose powders used. Single- and double-encapsulated powders had similar densities, but the total fat content differed. There was a 25% increase in total fat content in the double-encapsulated powder due to the added vegetable waxes. There was also a 14% increase in extractable fat in the double-encapsulated powder. However, percentages of total fat extracted were similar: 25.9% for the single- and 24.3% for the double-encapsulated powders. Physical properties such as fat content, moisture and product density affect powder flow (Peleg, 1977). Encapsulated material was affected less by these properties when the outer coat was composed of amorphous sucrose or other sugars. Double-coated powders were less free-flowing when the outer coat was a waxy vegetable matrix, because fat on the surface of powders has a tendency to cause the particles to adhere to one another or agglomerate (Onwulata et al., 1995).

The particle size distribution (Fig. 1) showed the sucrose and single-encapsulated powders ranged from 20 to 800  $\mu m$  in size, while the double-encapsulated powders ranged from 50 to 1300  $\mu m$ . The mean distribution showed that double-encapsulated powders (750  $\mu m$ ) were two times larger than the sucrose and single-encapsulated powders (350  $\mu m$ ). Larger sizes increase the potential for flow problems. The double-encapsulated powder had a normal Gaussian distribution. Single-encapsulated particles fell within a narrower particle size range, with relatively uniform distribution, but the double-encapsulated powders had a broader size range. Uniformity in particle size and shape improves powder flow (White et al., 1967). Previous work (Onwulata et al., 1994) with milkfat encapsulated in sucrose showed little or no milkfat present on the particle surface to interfere with flow. Therefore, flow difficulties with double-encapsulated powders were probably attributable to particle size and the waxy second coat.

Flow properties of the powders (Table 2) showed crystalline sucrose was free flowing ( $P < 0.1$ ), with flow increasing under mechanical shaking. Single-encapsulated powders were a log order of magnitude less free flowing (Konstance et al., 1995). Double-encapsulated powders were 50% less flowable due to their size and the waxy top coat. With encapsulated powders, 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 (Onwulata et al., 1994). Flowability of a powder is determined by both the physical properties of the powder and the geometry of the particles. Several experimental methods measure relative flow characteristics. Flow dynamics are influenced by particle density, bulk density, particle shape and size as well as composition (White et al., 1967).

Compressibility as a measure of internal cohesion and mechanical strength at various

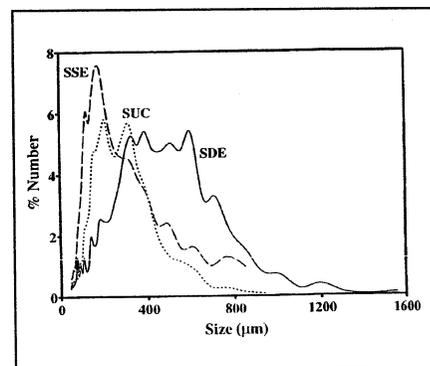


Fig. 1—Particle size distribution of sucrose (SUC), single-encapsulated (SSE) and double-encapsulated powders (SDE).

## Single and Double-Encapsulated Powders . . .

**Table 1—Physical properties of single- and double-encapsulated powders<sup>a</sup>**

	Moisture %	Loose density (g/cm <sup>3</sup> )	Bulk density (g/cm <sup>3</sup> )	Total fat %	Extractable fat %
Sucrose (granulated)	0.12±0.01	0.84±0.01	1.58±0.02	—	—
Single-encap	1.13±0.05	0.24±0.01	1.10±0.01	50.9±0.91	13.2±1.20
Double-encap	1.53±0.13	0.26±0.01	1.09±0.01	62.9±1.01	15.3±0.57

<sup>a</sup>—No fats present; ±standard deviation.

**Table 2—Flow and compressibility properties of single- and double-encapsulated powders<sup>a</sup>**

	G-glow (g/sec)	M-flow (g/sec)	% Compressibility <sup>b</sup>		
			100N	200N	300N
Sucrose (granulated)	190.0±3.2	228.6±1.6	4.7	6.9	7.4
Single-encap	71.5±6.0	72.2±6.0	25.5	32.5	35.0
Double-encap	*	36.6±3.2	36.3	43.6	50.9

<sup>a</sup>\*No flow. G-FLOW=gravitation flow; M-FLOW=mechanical agitation to enhance flow; ± standard deviations.  
<sup>b</sup>Preset compression force (N).

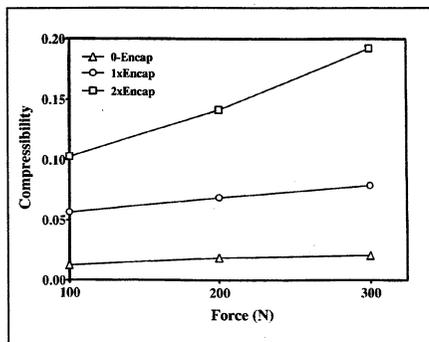
loads (Table 2) increased ( $P<0.05$ ) for single-encapsulated powders as well as ( $P<0.01$ ) for the double-encapsulated powders (Fig. 2). Compressibility increased with increased force depending on type of encapsulation and there was a concurrent increase in cohesiveness. Increased compression did not lead to powder caking or expulsion of encapsulated fat either in the single- or the double-encapsulated powder (data not shown). The sucrose powder was less compressible than its single- and double-encapsulated counterparts (Konstance et al., 1995). Carr (1976) had shown that compressibility, under relatively small loads, was a sensitive index of cohesiveness and could be used to predict potential flow problems.

SEM images of single-encapsulated powders (Fig. 3) showed the surfaces had irregular ridges with ovoid dimpling, and the particles were somewhat agglomerated (3A). The internal structures (3B) were consistent with our earlier results that showed unique micro-cavities uniformly distributed within the particle, without central voids (Onwulata et al., 1996). Surfaces resembled those described by Buma and Henstra (1971) for some spray-dried milk products and by Rosenberg and Young (1993) for milkfat encapsulated in whey proteins.

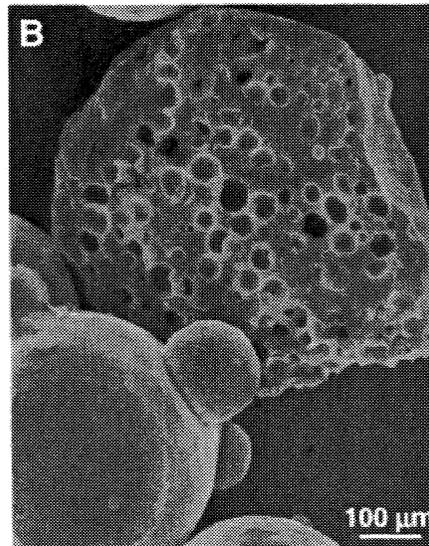
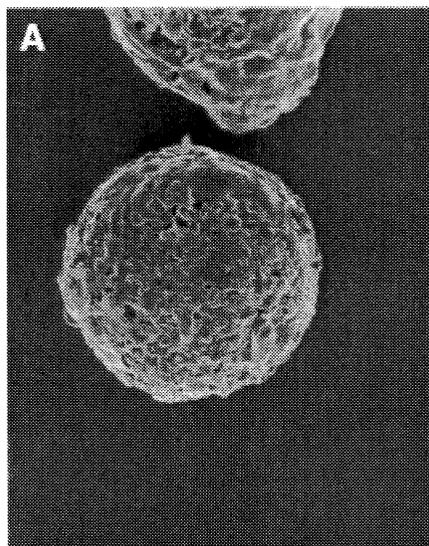
In contrast, the surface of the double-encapsulated powder, (Fig. 4A) was very smooth, indicating the effect of the double-coat. The fractured particle shows the enclosure of the single-encapsulated particle within the double-capsule (4B). The fine particle structure of the double-encapsulated surface (Fig. 5) shows a matrix formed with embedded hydrogenated stearine materials, and the

mesh of linear polymeric alcoholic materials. This shows that the process of manufacturing the double-encapsulated powders formed a waxy matrix around the powder particles, which led to the smooth surface coating. Within the double-coat, the single-encapsulated particles were as described earlier (Onwulata et al, 1995).

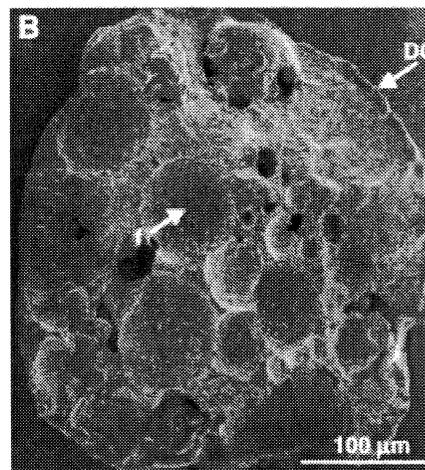
The mode of double-encapsulation (Fig. 6) is that either a single large particle (A) or several smaller particles (B) are encased



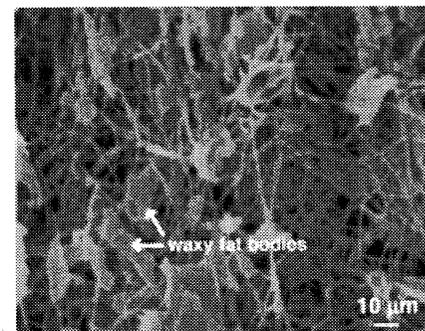
**Fig. 2—Relative compressibility of sucrose (0-Encap), single-encapsulated (1x-Encap) and double-encapsulated powders (2x-Encap).**



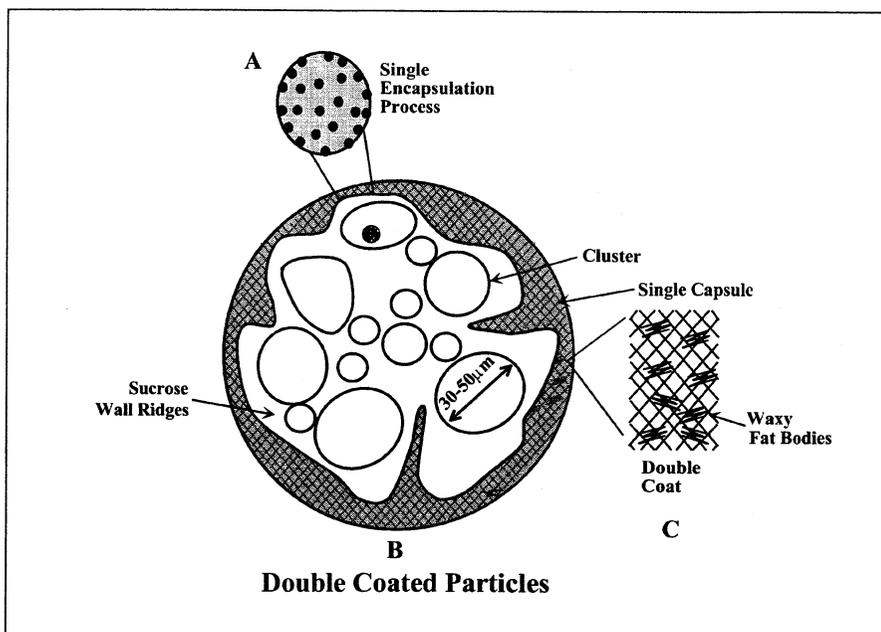
**Fig. 3—Scanning electron photomicrographs of intact (3A) and dry fractured (3B) particles containing 50% butteroil encapsulated in sucrose.**



**Fig. 4—Scanning electron photomicrographs of intact (4A) and dry fractured (4B) particles containing 50% butteroil encapsulated in sucrose and double-coated with vegetable waxes. (s): uneven, indented surface ridges. (DC): Double-coat. (f): Fat locations.**



**Fig. 5—SEM photomicrograph of the surface of a double-encapsulated powder (1000X).**



**Fig. 6—** Schematic depiction of the mode of double-encapsulation of butteroil. (A): Intact single-encapsulated particle. (B): Single-encapsulated particles encapsulated with the double-coat. (C): Matrix of the surface of the double-coat.

within the matrix of the double-encapsulated powder (C). Therefore, a double-coated powder could be a simple or compound powder with more than one particle encapsulated. This helps to explain the wide distribution in particle sizes (50 to 1200 m). There are many possibilities for double-encapsulated powders, especially considering the large number of compounds that could be used as the second coat.

The moisture uptake rates of the different powders (Fig. 7) fit an exponential function, the rate of uptake being dependent on the nature of the powder and exposure conditions. Sucrose is highly hygroscopic and its moisture uptake was higher than that of the single-encapsulated powder. Further reduction in moisture was shown with the double-encapsulated powder. In terms of moisture sorption or as a moisture barrier, the double-encapsulated powder was more efficient. This functionality enhances the benefits of encapsulation, and may ameliorate or eliminate the need for special packaging to prevent moisture uptake during storage (Onwulata and Holsinger, 1995).

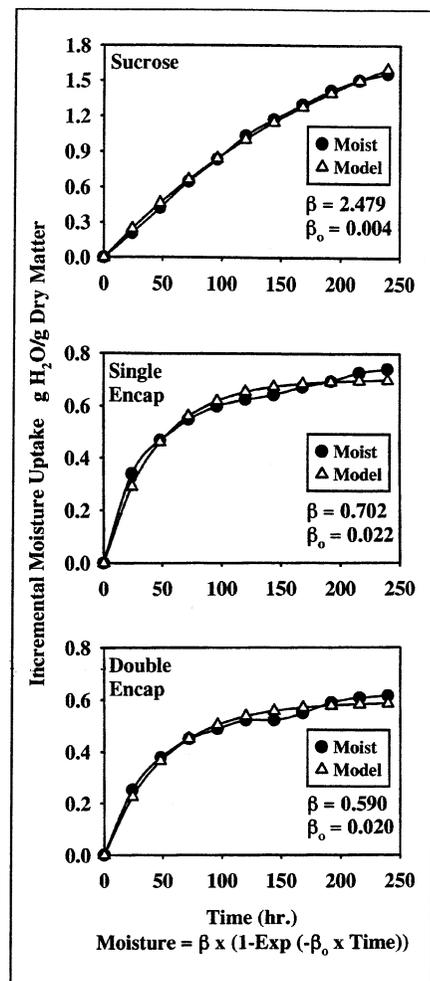
## CONCLUSIONS

DOUBLE-ENCAPSULATED POWDERS HAD properties that differed notably from those of the single-encapsulated powders or crystalline sucrose. Large particle size, lower density and waxy surfaces impeded powder flow. However, the double-encapsulated powders had higher resistance to moisture sorption. Through choice of wall material (such

as sucrose) and the powder preparation methods described here, an effective carrier for sensitive ingredients may be realized.

## REFERENCES

- Andersen, S. 1995. Microencapsulated marine omega-3 fatty acids for use in the Food Industry. *Food Tech Europe*. Dec. 1994/Jan. 1995: 104-106.
- Anonymous. 1978. Analytical methods for dry milk products. Niro Atomizer Co., Copenhagen, Denmark.
- AOAC. 1984. *Official Methods of Analysis*, 14th ed. Association of Official Analytical Chemists, Washington, DC.
- Buma, T.J. and Henstra, S. 1971. Particle structure of spray-dried milk products as observed by a scanning electron microscope. *Neth. Milk and Dairy J.* 25:75-80.
- Carr, R.L. 1976. Powder and granule properties and mechanics. In *Gas-Solids Handling in the Processing Industries*, J.M. Marchello and A. Gomezplata (Ed.). Marcel Dekker, Inc., New York.
- Duxbury, D.D. and Swientek, R.J. 1992. Encapsulated ingredients face healthy future. *Food Processing*. Feb: 38-46.
- Flink, J. and Karel, M. 1970. Effects of process variables on retention of volatiles in freeze-drying. *J. Food Sci.* 35: 444-447.
- Jackson, L.S. and Lee, K. 1991. Microencapsulation and the food industry. *Lebensm.-Wiss. u. -Technol.* 24: 289-297.
- Konstance, R.P., Onwulata, C.I., and Holsinger, V.H. 1995. Evaluation of flow properties of spray-dried encapsulated butteroil. *J. Food Sci.* 60:841-844.
- Menting, L.C. and Hoagstad, B.J. 1967. Volatiles retention during the drying of aqueous carbohydrate solutions. *J. Food Sci.* 32: 87-90.
- Moreyra, R. and Peleg, M. 1980. Compressive deformation patterns of selected food powders. *J. Food Sci.* 45: 864-868.
- Onwulata, C.I., Smith, P.W., Craig Jr., J.C., and Holsinger, V.H. 1994. Physical properties of encapsulated spray-dried milkfat. *J. Food Sci.* 59: 316-320.
- Onwulata, C.I. and Holsinger, V.H. 1995. Thermal properties and moisture sorption isotherms of spray-dried encapsulated milkfat. *J. Food Proc. Pres.* 19: 33-51.
- Onwulata, C.I., Smith, P.W. and Holsinger, V.H. 1995. Flow and compaction of spray-dried powders of anhydrous butteroil and high melting milkfat encapsulated in disaccharides. *J. Food Sci.* 60: 836-840.
- Onwulata, C.I., Smith, P.W., Cooke, P., and Holsinger, V.H. 1996. Particle structures of encapsulated milkfat



**Fig. 7—** Moisture uptake of sucrose and single- and double-encapsulated powders.

- powders. *Lebensm.-Wiss. u. -Technol.* 29: 163-172.
- Peleg, M. 1977. Flowability of food powders and methods for its evaluation - a review. *J. Food Proc. Eng.* 1: 303-328.
- Pothakamury, U.R. and Barbosa-Canovas, G.V. 1995. Fundamental aspects of controlled release in foods. *Trends in Food Sci. & Technol.* 6: 397-406.
- Redding, B.K. 1990. Apparatus and method for making microcapsules. U.S. Patent. 4,978,483.
- Redding, B.K. 1993. Method for inducing transformations in waxes. U.S. Patent 5,209,879.
- Rockland, L.B. and Nishi, S.K. 1980. Influence of water activity on food product quality and stability. *Food Technol.* 34(4): 42-59.
- Rosenberg, M. and Young, S.L. 1993. Whey proteins as microencapsulating agents. Microencapsulation of anhydrous milkfat-structure evaluation. *Food Microstructure* 12: 31-41.
- Sone, T. 1972. *Consistency of Foodstuff*, p. 133-142. D. Reidel Publishing Co., Dordrecht, The Netherlands.
- White, G.W., Bell, A.V., and Berry, G.K. 1967. Measurement of the flow properties of powders. *J. Fd. Technol.* 2: 45-52.
- Wagner, L.A. and Warthesen, J. J. 1995. Stability of spray-dried encapsulated carrot carotenes. *J. Food Sci.* 60:1048-1053.
- Ms received 6/6/97; revised 9/10/97; accepted 9/15/97.

The assistance of Mr. Jerome Harden of Delta Food Groups, Inc. with the double-encapsulation process, and the work of Dr. Peter Cooke, Mr. Richard Stoler, Mr. Lenier Tucker and Mr. Rashid Burwell are greatly appreciated.

Mention of a brand name or firm does not constitute an endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.