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Flow Properties Of Encapsulated Milkfat Powders as Affected by Flow Agent

C.I. ONWULATA, R.P. KONSTANCE, and V.H. HOLSINGER

ABSTRACT

'ood powders containing softer cores of fatty or flavor components present handling problems, as such powders are generally of very low density and mostly non free-flowing. Three classes of flow conditioners [silica, silicate and stearate], at three levels of concentration, were evaluated to determine effects on bulk and flow properties of sucrose, lactose and modified corn starch as well as butter oil powders encapsulated with the same materials. Silica and silicate were generally effective in improving bulk properties but only silica was effective in improving flowability of unencapsulated powders as well as lactose-encapsulated butteroil powder. Washing with isopropanol was effective in improving flow only with the lactose-encapsulated butteroil powder. Butteroil encapsulated in lactose had flow characteristics similar to lactose.

Key Words: encapsulation, butteroil, flowability, milk powder

INTRODUCTION

ENCAPSULATION may lead to difficulties in handling due to changes in bulk properties of the powders. This is particularly true for encapsulated butteroil where the encapsulated powders show a propensity for stickiness and lumping. As the need for these and other delicately designed food powders with flavorful cores increase, the need for methods to improve their flow and handling characteristics becomes increasingly important (Konstance et al., 1995).

Flow problems result from inter/intra-particular forces, powder particle size and shape as well as moisture and fat content. Conditioners (or anti-caking agents) enhance powder flow by reducing some interparticle forces reducing cohesiveness and compressibility while increasing bulk density (York, 1975; Peleg and Mannheim, 1973). Several classes of chemical agents have been used as flow conditioners. These include silicates, stearates and phosphates and are relatively effective depending on the mode of particle friction in the food powders. The mode of action of most flow agents is to separate particles, making them more free flowing (Peleg and Hollenbach, 1984).

Adding flow conditioners changes the bulk properties making them more complex. Mixed food powders have different densities and compressibilities. Some powders with added flow conditioners tend to behave as single component powders because the smaller particles adhere to surfaces of larger particles. Barbosa-Canovas et al. (1987), showed the density and compressiblity of mixtures of powders were dependent on particle sizes. With encapsulated powders, the addition of flow conditioners may further complicate flow behavior.

Powder compaction is sometimes described by a formula which relates the ratio of tapped bulk density to the loose bulk density (the "Hausner ratio," Hausner, 1967). The relation gives an indication of the internal friction index, mostly for non-cohesive powders. Using tap density, the magnitude of internal friction of powder particles gives an inference on the compaction and compressibility of the powder under low mechanical load (Grey and Beddow, 1969). Our objective was to investigate the effect of flow conditioners added at different concentrations on bulk density, flow and mechanical properties of lactose, sucrose and modified corn starch as well as butteroil encapsulated in those same materials.

MATERIALS & METHODS

THREE FLOW CONDITIONERS, calcium stearate (CS) and aluminum silicate (AS) (Penta Manufacturing, Fairfield, NJ) and silica (SS) (Sylox®, W.R. Grace, Baltimore, MD) were added at 0, 1 and 2% to powders of butteroil encapsulated with crystalline sucrose, lactose or modified corn starch (MCS) (CAPSUL®, National Starch and Chemical Co., Bridgewater, NJ) or to the encapsulating agents alone. Butteroil was obtained from a commercial manufacturer (Land-O'-Lakes, Inc., Arden Hills, MN). Spray dried powders containing 40% butteroil were prepared in our pilot plant as previously described (Onwulata et al., 1994). The moisture content for both lactose and sucrose was <1%, modified corn starch contained 5% moisture, and the spray dried encapsulated powders had 2–4% moisture. All powders were sieved mechanically to pass through a 500 μm sieve. The flow conditioners were added after sieving.

Powder was allowed to flow through a conical funnel at orifice diameters of sufficient size to barely permit flow. The angle of repose (θ), a measure of relative flowability of a given powder, was calculated from the base angle formed by the heap of powder (Sjollema, 1963). The mass flow of the powder (g/sec) was measured by permitting 80 g to flow through funnels of outlet diameter 0.5–3.0 cm with gentle shaking (FMC/Synthron, Homer, PA) at 40 rpm. Flow through the funnel (g) was divided by the orifice area (πr^2) to derive the flow area.

Encapsulated powders were washed in isopropyl alcohol to extract surface fat. External fat on the surfaces of the powder particles may act as a bridge between particles, so washing the surface fat from the particles might improve flow. Ten g of the powder were washed with 50 mL isopropyl alcohol with continuous shaking for 15 min. The powder and solvent were filtered, the extract containing fat was dried and percent fat calculated (Onwulata et al., 1994).

The loose bulk density (ρ_L) was determined from the weight of a given amount of powder and the known volume of the cell. The sample cell, as described by Moreyra and Peleg, (1980), was 30 mm high and 45 mm in diameter. The powders were compressed in the sample cell mounted on the base plate of a model 4200 INSTRON Universal Testing Machine (Instron, Canton, MA) at a crosshead speed of 10 mm/min using a 50 KG load cell to a preselected force of 40 kg. Absorbed energy and irrecoverable work were determined as described by Moreyra and Peleg (1980).

Powder compressibility was determined by evaluating the slope of the relationship between bulk density and corresponding compressive stress ($1 < \log \sigma < 4$) using

$$\rho_D = a + b \log \sigma; \text{ (Malave et al., 1985)} \tag{1}$$

where ρ_D = bulk density (g/cm³) at corresponding σ; σ = compressive stress (g/cm²); and a,b = empirical constants with "b" representing compressibility. Average Coefficient of Variation (CV) for compressibility was 3%.

Stress relaxation of the powders, which can be considered an index of the "solidity" of a compressed sample, was evaluated using the INSTRON by compressing a given powder to the same strain used for its compressibility analysis and measuring the stress relaxation over 5 min. The stress relaxation data were fitted to the form of Peleg (1979) using

$$(F_o t)/(F_o - F_t) = k_1 + k_2 t \tag{2}$$

where F_o = initial force (g); F_t = force (g) at time t (sec); and k_1,k_2 = constants with slope k_2 used as the "solid" index or solidity.

Tapped density (ρ_T) was determined by measuring the density of the powders after "hand-tapping" the container 300 times at 60 taps/min.

Table 1—Bulk density of powders

	Loose bulk density (G/CM^3)							Tapped bulk density (G/CM^3)							Hausner ratio[a]						
		Silica conc (%)		Silicate conc (%)		Stearate conc (%)			Silica conc (%)		Silicate conc (%)		Stearate conc (%)			Silica conc (%)		Silicate conc (%)		Stearate conc (%)	
Powder	0	1	2	1	2	1	2	0	1	2	1	2	1	2	0	1	2	1	2	1	2
Sucrose	0.81	0.82	0.80	0.85	0.84	0.86	0.81	0.92	0.91	0.85	0.91	0.91	0.77	0.78	1.14	1.11	1.06	1.07	1.08	0.90	0.96
Lactose	0.67	0.75	0.78	0.75	0.82	0.78	0.86	1.01	1.00	0.90	0.89	0.97	0.90	0.92	1.51	1.33	1.15	1.19	1.18	1.15	1.07
MCS[b]	0.37	0.53	0.42	0.46	0.47	0.44	0.43	0.76	0.65	0.73	0.61	0.62	0.58	0.55	2.05	1.23	1.74	1.33	1.32	1.32	1.28
Sucrose-Encapsulated Butteroil	0.33	0.43	0.32	0.37	*	0.36	*	0.55	0.55	0.70	0.54	*	0.51	*	1.67	1.28	2.19	1.46	*	1.41	*
Lactose-Encapsulated Butteroil	0.24	0.40	0.29	0.38	0.29	0.36	0.27	0.55	0.57	0.49	0.56	0.59	0.53	0.52	2.29	1.43	1.69	1.47	2.03	1.47	1.93
MCS-Encapsulated Butteroil	0.33	0.33	0.36	0.31	0.30	0.30	0.29	0.43	0.45	0.47	0.46	0.45	0.45	0.44	1.30	1.37	1.31	1.48	1.50	1.50	1.52
	PSD = 0.10[c] df = 266[d]							PSD = 0.0096 df = 342													

* Lumpy samples - No Measurements.
[a] Hausner Ratio = Tapped density/Loose density.
[b] MCS = Modified Corn Starch.
[c] PSD = Pooled Standard Deviation.
[d] df = Degrees of Freedom.

Table 2—Bulk properties of powders

	Compressibility (cm^{-1})							Irrecoverable work							Solidity						
		Silica conc (%)		Silicate conc (%)		Stearate conc (%)			Silica conc (%)		Silicate conc (%)		Stearate conc (%)			Silica conc (%)		Silicate conc (%)		Stearate conc (%)	
Powder	0	1	2	1	2	1	2	0	1	2	1	2	1	2	0	1	2	1	2	1	2
Sucrose	0.03	0.10	0.03	0.02	0.3	0.03	0.03	84.7	68.9	82.3	76.8	82.4	81.2	84.9	3.5	1.5	3.8	3.0	3.2	3.7	2.0
Lactose	0.07	0.05	0.04	0.05	0.05	0.06	0.04	90.4	86.3	85.9	82.1	84.1	86.7	81.4	5.7	4.1	5.8	8.3	8.9	3.9	3.1
MCS[a]	0.10	0.07	0.05	0.10	0.10	0.11	0.11	84.9	79.8	84.9	85.6	84.3	90.9	92.2	3.4	2.9	2.6	2.8	3.1	2.8	
Sucrose-Encapsulated Butteroil	0.10	0.06	0.03	*	*	*	*	96.4	92.7	92.8	*	*	*	*	2.8	1.7	2.8	*	*	*	*
Lactose-Encapsulated Butteroil	0.08	0.08	0.05	0.12	0.16	0.12	0.13	91.2	90.3	90.7	91.0	95.0	89.1	95.5	5.5	5.0	5.1	6.6	6.6	5.9	3.8
MCS-Encapsulated Butteroil	0.10	0.06	0.06	0.11	0.10	0.10	0.10	87.6	88	87.8	87.1	87.8	87.2	85.6	3.2	3.3	2.7	3.5	2.8	3.9	3.0
	PSD = 0.002[b] df[c] = 114							PSD = 1.80 df = 114							PSD = 0.45 df = 114						

* Lumpy samples - No Measurements.
[a] MCS = Modified Corn Starch.
[b] PSD = Pooled Standard Deviation.
[c] df = Degrees of Freedom.

Density determinations (g/cm^3) were made in triplicate. Compaction of powders was described by the ''Hausner ratio''. Statistical analyses were performed using the General Linear Methods (GLM) procedure (SAS Institute, Inc., 1989). Significant correlations among variables were determined by the Proc CORR subroutine (SAS Institute, Inc., 1989). All samples were evaluated and results reported as averages of four measurements.

RESULTS & DISCUSSION

BULK PARAMETERS, such as density and compressibility are sensitive indices to changes in powders (Hollenbach et al., 1982). The effects of flow conditioner concentration were compared on the loose densities of all powders (Table 1). Typically, encapsulated powders were less dense than encapsulating agents alone because of the butteroil component and the increase in particle size. The increase in loose density that accompanies the expected reduction of interparticle forces was observed with the addition of flow conditioner at 1% in all powders except MCS where loose density was effectively unaltered. Addition of higher concentrations of flow conditioner resulted in a flat response or a reduction in loose densities as reported previously (Nash et al., 1965, Hollenbach et al., 1983). The most notable increase in loose density was observed with the addition of 1% silica to the butteroil powders encapsulated in lactose. The addition of silicate and stearate at the 2% level to the butteroil powders encapsulated in sucrose caused considerable agglomeration and densities were not determined. Changes in bulk density as a result of tapping were compared (Table 1). Large increases in bulk density resulting from tapping or compaction are indicative of potential flow problems. The Hausner Ratio (Hausner, 1967) is frequently used as a relative flowability index.

The remaining bulk parameters (compressibility, irrecoverable work and solidity) were also compared (Table 2). Compressibility in many powders is a measure of internal cohesion, flowability, and to some extent, deformability. A lower compressibility is usually indicative of a less cohesive powder and one that has greater bulk density. Each of the flow conditioners was effective in reducing compressibility of the unencapsulated powders when applied at 1% concentration. The compressibility of the sucrose samples was similar to that observed for the density measurements in that best flow was achieved at 1% added flow conditioner. Generally, the compressibility of the remaining unencapsulated powders continued to decrease with added flow conditioner. The only effective additive for the encapsulated powders was silica (2%) which resulted in a 35 to 70% decrease in compressibility.

The amount of energy absorbed as a result of compression (or irrecoverable work) also provides an index of powder cohesiveness. The work required to compress a relatively non-

Table 3—Flow properties of powders

	Repose Angle (Degrees)							Mech Flow Rate[a] (G/SEC)							Change in Bulk Density (%)						
		Silica Conc (%)		Silicate Conc (%)		Stearate Conc (%)			Silica Conc (%)		Silicate Conc (%)		Stearate Conc (%)			Silica Conc (%)		Silicate Conc (%)		Stearate Conc (%)	
Powder	0	1	2	1	2	1	2	0	1	2	1	2	1	2	0	1	2	1	2	1	2
Sucrose	58.7	59.9	58.2	58.6	57.8	59.2	57.6	152.1	169.0	188.2	162.1	158.2	141.4	144.6	NA[d]	1.2	−1.2	4.9	3.7	6.2	0.0
Lactose	50.1	54.9	53.9	51.7	53.8	55.7	47.5	41.5	52.3	51.3	77.0	69.0	54.3	50.5	NA	11.9	16.4	11.9	22.4	16.4	28.4
MCS[b]	44.4	65.9	60.7	51.7	5.4	44.7	46.3	27.3	153.4	153.0	104.9	102.8	28.5	21.8	NA	43.2	13.5	24.3	27.0	18.9	16.2
Sucrose-Encapsulated Butteroil	45.0	43.4	45.4	49.2	50.3	42.9	—[c]	22.7	8.8	—	—	—	—	—	NA	30.3	−3.0	12.1	*[e]	9.1	*
Lactose-Encapsulated Butteroil	55.1	55.2	57.6	55.1	55.1	53.5	50.5	10.5	49.4	44.3	34.6	43.9	8.0	7.0	NA	66.7	20.8	58.3	20.8	50.0	12.5
MCS-Encapsulated Butteroil	50.0	62.6	62.9	53.0	51.8	53.5	54.9	88.1	38.0	40.9	19.0	15.1	20.3	20.1	NA	0.0	9.1	−6.1	−9.1	−9.1	*
	PSD[f] = 1.98 df[g] = 123							PSD = 0.38 df = 111													

[a] Flow Rate flow through 30 mm orifice.
[b] MCS = Modified Corn Starch.
[c] No results.
[d] NA = Not Applicable, Data used for calculation.
[e] Lumpy samples - No Measurements.
[f] PSD = Pooled Standard Deviation.
[g] df = Degrees of Freedom.

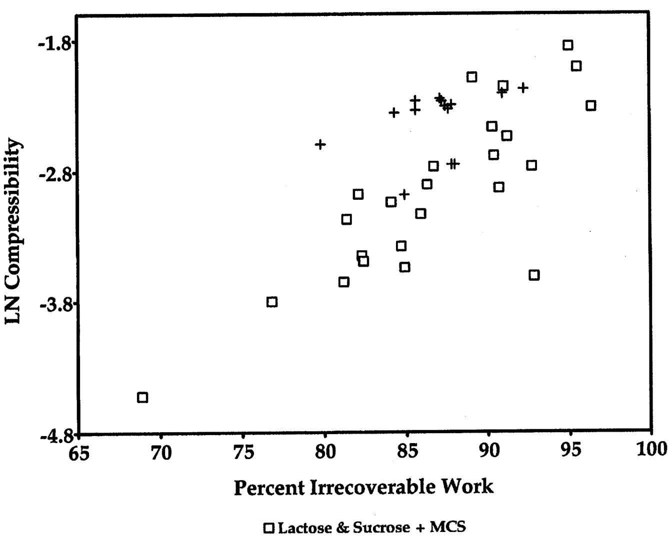

Fig. 1—Compressibility as related to Irrecoverable work of unencapsulated and butteroil-containing powders encapsulated in carbohydrates. □ Lactose and Sucrose, +Modified Corn Starch (MCS).

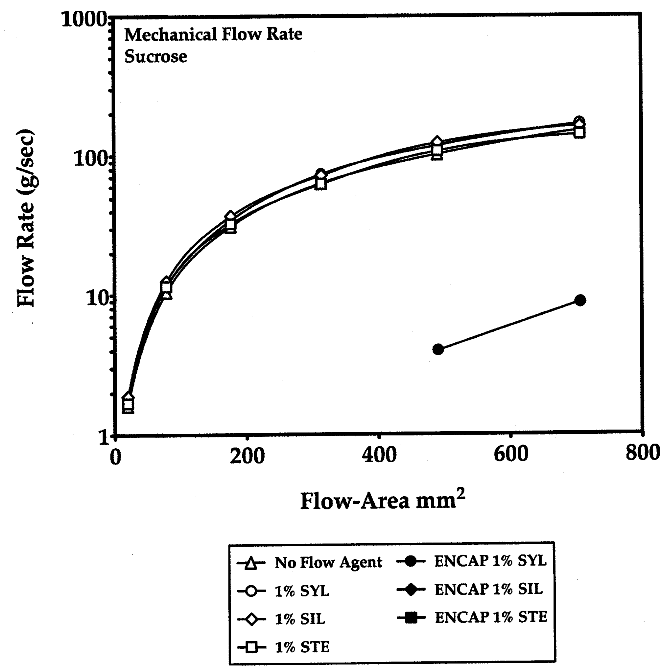

Fig. 2—Flow distribution of sucrose with added flow agents: SYL = Silica, SIL = Silicate, STE = Stearate, ENCAP = Butteroil encapsulated with sucrose; Mechanical flow.

cohesive powder is mostly recoverable (Moreyra and Peleg, 1980). The irrecoverable work was plotted as a function of compressibility (Fig. 1). A strong positive correlation ($r^2 = 0.821$) existed for the curves of the lactose and sucrose powders, both unencapsulated and containing butteroil. The data were described by the empirical equation:

$$\ln C = 0.91e^{-10.85W} \tag{3}$$

where C = compressibility and W = irrecoverable work.

Compressibility of the MCS powders was essentially insensitive to irrecoverable work. The modified corn starch is elastic and recovered much closer to its natural state after compression.

In the case of unencapsulated powders, each of the flow conditioners (at the 1% level) was effective in reducing irrecoverable work. Increasing the concentration of flow conditioner generally resulted in an increase in absorbed energy. Studies by Peleg et al. (1973) showed that as concentrations of stearate or silicate (added to sucrose) were increased from 1 to 3%, there was a reduction in cohesiveness by 1 to 2% but cohesiveness increased as more flow conditioner was added.

Evaluation of the solid nature of the powders (Table 2) showed differences where larger values for solidity imply a more elastic and less cohesive, compacted material. The silicate flow conditioner appeared to be most effective in achieving an increased solid nature and this was especially true for lactose powders, both unencapsulated and containing butteroil.

The effect of flow conditioner was compared on the flow properties of various powders (Table 3). The addition of silica and silicate seemed to provide a slight improvement in unencapsulated sucrose flow rate (mechanical flow through a 30 mm orifice) but had little or no effect on the repose angle. Flow rates of the butteroil-encapsulated-in-sucrose powders were considerably or completely retarded by addition of any of the flow conditioners.

A slight increase in flow of the unencapsulated lactose powder was observed with the addition of each of the flow conditioners. However, as observed with sucrose, very little effect on

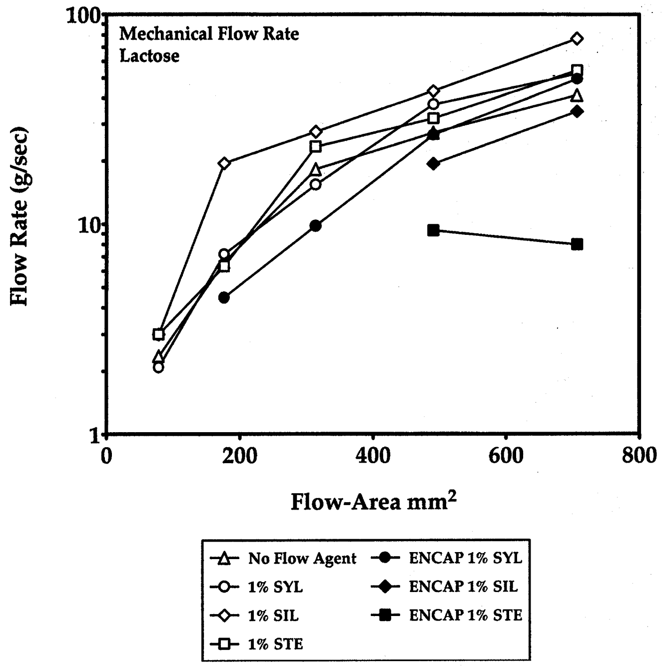

Fig. 3—Flow distribution of lactose with added flow agents: SYL = Silica, SIL = Silicate, STE = Stearate, ENCAP = Butteroil encapsulated with lactose; Mechanical flow.

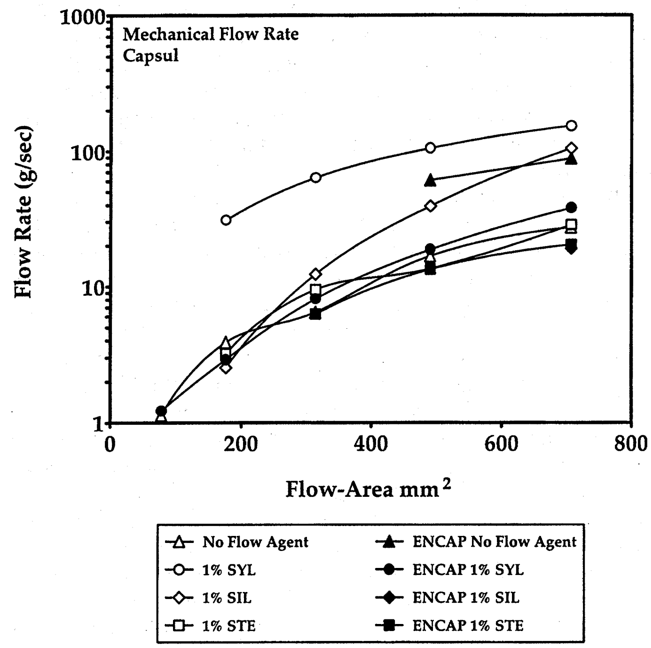

Fig. 4—Flow distribution of modified corn starch with added flow agents: SYL = Silica, SIL = Silicate, STE = Stearate, ENCAP = Butteroil encapsulated with modified corn starch; Mechanical flow.

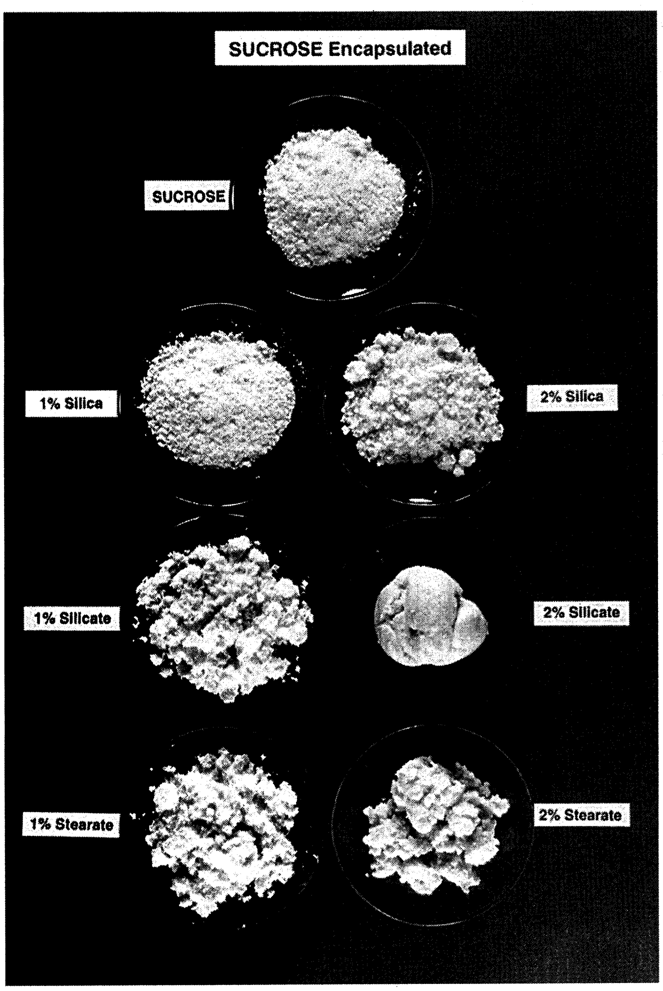

Fig. 5—Butteroil-containing powders encapsulated in sucrose, showing stickiness and lumping with added flow agents.

Table 4—Properties of butteroil-containing powders encapsulated in sucrose, lactose on modified corn starch washed with isopropanol

	Loose density (g/cm^3)	Tapped density (g/cm^3)	Compressability (cm^{-1})	Irreversible work (%)	Solid	Repose angle (degrees)	Mechanical flow (g/sec)
SUCROSE							
Control	0.33	0.55	0.10	96.4	2.76	45.0	22.7
Washed	0.43	0.67	0.17	95.4	2.89	50.6	—
LACTOSE							
Control	0.24	0.55	0.08	91.2	5.47	55.1	10.5
Washed	0.31	0.45	0.04	94.6	3.41	52.9	24.2
MCS[a]							
Control	0.33	0.43	0.10	87.6	3.16	50.0	88.1
Washed	0.27	0.38	0.08	87.5	3.72	54.0	28.7

[a] MCS = Modified Corn Starch.

improving the repose angle resulted, but the angle slightly increased. The butteroil powders encapsulated in lactose however, showed notable improvement in flow with addition of either silica or silicate.

The unencapsulated starch had a very low flow rate to begin with and addition of silica or silicate notably improved the flow whereas the stearate had little or no effect on flow (Table 3). The butteroil powders encapsulated in starch, with no added flow agent, flowed more readily than unencapsulated starch. The addition of flow conditioners had a negative effect and flow was retarded in all cases.

The repose angles of each of these powders, both unencapsulated and containing butteroil indicated either cohesive or very cohesive materials as categorized by Peleg (1983) and we expected limited flowability. The repose angles in this case, however, were not good indicators of change in flowability resulting from the flow conditioner. At this magnitude (45–65 degrees) of angle of repose, the relatively small changes were probably overcome, and perhaps confounded, by the irregular shapes of the heaps and impact velocity. A more effective indicator of change in flowability from our study was the evaluation of the change in loose density of the powders when flow conditioner was added. Peleg (1983) studied the effectiveness of flow conditioners and the need for a surface affinity between host particles and the flow agent. When the surface affinity was inadequate, as indicated by small changes in loose density (i.e. < 10%), the particles of flow conditioner may segregate and, rather than reducing interparticle forces, would fill interparticle spaces. The change in loose density as a result of added flow conditioner was compared (Table 3) and, using the 10% increase as a rule of thumb, it was generally effective in predicting flow improvement.

The classic increase in bulk density with tapping was noted for all powders (Table 1). The bulk density after "n" number of taps depended on loose bulk density (ρ_L). Flowability was characterized using the Hausner ratio (ρ_L/ρ_T). Generally as ratio increased, the flow rate decreased. These data were in accordance with the flow indications represented by the change in loose density and accurately reflected that particle friction was a factor in retarding flow (Grey and Beddow, 1969).

The log plots of flow rate vs flow area (Fig. 2, 3, 4) show the encapsulated butteroil powders with flow rates that were an order of magnitude less than those for the unencapsulated powders. The shape of the curve is intuitive where small and restrictive flow areas considerably reduced powder flow and logarithmic changes followed. The behaviors of unencapsulated powders were all very similar with order of flowability being sucrose > MCS > lactose. The effect of the admix of flow conditioners in rupturing particle walls of the butteroil powders encapsulated in sucrose was studied (Fig. 5). The addition of silica had a minimal effect whereas the addition of either silicate or stearate led to structural damage, release of fat, lumping and eventual complete retardation of flow.

Encapsulated butteroil powders were washed in isopropyl alcohol to remove any surface fat that may have contributed to the low flow (Table 4). The alcohol treatment increased the bulk density of the butteroil powders with sucrose and lactose as encapsulating agents only slightly. The most notable effect was observed with butteroil powder encapsulated in lactose where reductions in tapped density and compressibility resulted as well as an increase in powder flowability. The amounts of milk fat extracted as a result of this washing step were: sucrose—3.83%, MCS—4.37%, and lactose—5.77% of the total fat. Although the major factor in flow retardation was product density, surface fat contributed to reduced flow, especially in the butteroil-containing powders encapsulated in lactose.

CONCLUSION

THE ADDITION OF SILICA AND SILICATE as flow conditioners were effective in improving flowability of unencapsulated powders as well as butteroil-containing powders encapsulated in lactose. The best concentration of flow conditioners seemed to be 1% under our experimental conditions, with little or no further improvment thereafter. The addition of 1% silica to the butteroil powder encapsulated with lactose led to flow behavior similar to that of unencapsulated lactose. Bulk properties of loose bulk density, compressibility, tapped density (Hausner ratio) and irrecoverable work were adequate indicators of flow behavior. The stearate flow conditioner resulted in flow retardation of all powders studied. An isopropanol wash to remove surface fat was effective in slightly improving the flow of butteroil-containing powders encapsulated in lactose.

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Mention of a brand or firm name does not constitute endorsement by the USDA over others of a similar nature not mentioned.