Agglomeration of Milk Powder and Its Influence on Reconstitution Properties

Abstract
Powder granulation involving rewetting, granulation, drying, and screening has been used to simulate commercial instantizing of nonfat dried milk (NFDM). Effects of rewetting moisture on dispersibility, sinkability, solubility, bulk density, and porosity of the redried granulated product have been studied, with effects of particle size on reconstitution. Rewetting moisture is most critical in actual granule formation, a sharp break point occurring at 11-12% moisture, and optimum conditions for reconstitution coinciding with this point. Granulation increased dispersibility of powder from 41 to 62% at optimum rewetting conditions, whereas beyond the break point there occurred a sharp decrease in dispersibility and solubility. Optimum particle or granule size for the reconstitution has been determined by sieve fractionation of the granulated product. Particle size of 200 µ represented optimum dispersibility associated with infinite sinkability. Two major variables influencing reconstitution of instantized NFDM are rewetting moisture and particle size of the redried product.

Enlargement or agglomeration operations used in many industries. One operation is that of wet granulation used in the pharmaceutical and fertilizer industries.

Undoubtedly, much work has been done on the agglomeration of food powders and, in particular NFDM, by instantizing processes. Unfortunately little has been published, as most instantizing processes are patented. Nevertheless, the paucity of information indicates the difficulty in conducting controlled experiments in commercially available instantizing equipment.

The rewetting moisture content during agglomeration is acknowledged as commercially important, but the range of rewetting moistures specified in patents shows great variation, and appears to be the basis of differences between patents. For example, Peebles (22) specified an optimum rewetting moisture of 15%; Louder and Hodson (15) an optimum of 5.5%; and Scott (24) specified 10-14% rewetting moisture. Processes for instantizing milk powders are described by Bullock (5).

Peck (21) and Little and Mitchell (14) describe wet granulation practices in the pharmaceutical industry, giving no quantitative moisture requirements.

More fundamental research has been done on the mechanism of granule formation from moist powdered material, such as moist sand, in a tumbling-type drum mixer by Newitt and Conway-Jones (20) and Capes and Danckwerts (6). Newitt and Conway-Jones (20) describe three states of water in an assembly of spherical particles, viz., pendular, funicular, and capillary state. The capillary state is a saturation of the voids between particles with liquid. These workers postulate that in granulation a kneading action occurs, resulting in decreasing internal pore space; hence, if sufficient water is present the pores may become saturated to form stable granules. Capes and Danckwerts (6) extended this work and showed that in granulation of sand significant growth of granules occurred only when liquid contents were equal to between 90 and 110% of the amount required to fill the voids in a highly compacted sample. Saturation of voids in the

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aggregated particles with liquid is necessary to obtain a stable system.

Another important factor affecting reconstitution characteristics of powder is particle size. However, conclusive evidence is lacking concerning the optimum particle size for milk powder as revealed by King (12).

Bockian et al. (4) noted that instant powders consisted of very large aggregates of 180-840 μ, and suggested that this may be one of the main factors affecting dispersibility of instant NFDM. Similarly, Peebles (22) considered it important that approximately 80% of instant NFDM have particle sizes greater than 74 μ, to obtain a powder of desired characteristics.

Swanson (25) fractionated powders into different sizes and concluded that NFDM particles of 30-50 μ in diameter seemed to wet and disperse most readily, whereas, sizes on either side showed decreased dispersibility. Baker and Bertok (2) sieve-fractionated powders and concluded that decreased particle size was associated with decreased wettability and dispersibility.

Hall and Hedrick (9) stated that manufacturers of instant NFDM are fully aware of the importance of particle size. They describe a desirable particle distribution as being in the range 100-500 μ; no more than 10-15% of weight of particles should be less than 150 μ in diameter. Similarly, Claus and Brooks (7) dealt with the properties of instantized wheat flours, emphasizing increased particle size.

Researchers have shown the importance of particle size on dispersion of a food powder, although there appears to be little evidence for specifying an optimum size, even for NFDM. Pyne and Coulter (23) emphasized that there must exist an optimum size corresponding to maximum dispersibility. They found that dispersion of milk powders is inversely proportional to the surface-to-mass ratio while the rate of solution is directly proportional to this ratio. It is reasonable to deduce, therefore, that an optimum surface-to-mass ratio, or particle size, exists, and is related to optimum reconstitution characteristics.

It was the aim of our study to simulate an agglomeration process, applying the principles of wet granulation. Once a simulated instantizing process has been developed, it can then readily be employed to study some of the basic factors controlling the process; such as, effects of rewetting moisture and of particle size on the reconstitution properties of NFDM.

### Experimental Procedure

**Dispersibility.** The dispersibility of the NFDM powders and granules was determined by a modification of the American Dry Milk Institute (ADMI) method, described by Neff and Morris (19). Dispersibility is expressed as the percentage of powder reconstituted under conditions of test.

**Sinkability index.** The sinkability index of a powder was determined by the method of Neff and Morris (19), being expressed as mg of powder sinking per minute per cm² of area.

**Solubility index.** This was determined by the ADMI method (1), being expressed as ml of sediment.

**Bulk density, particle density, and porosity.** These properties of a powder were determined by a hexane displacement method, described by Beckett et al. (3). The method measures the bulk density and porosity of a loosely compacted sample. Bulk density and particle density are expressed as g/cc, while porosity is expressed as cc of voids per cc of bulk powder.

**Moisture.** Moisture was determined by drying weighed samples in a fan-assisted air oven at 102-103 C for three hours. Moisture was expressed on a wet weight basis.

**State of lactose in NFDM.** The milk powder was examined for crystalline lactose by polarized light microscopy, as described by King (11).

**Sieve separation.** Sieving of samples was done using British Standards test sieves, together with a mechanical sieve vibrator. All powder handling and sieving was done in a room held at a relative humidity of 40%, to minimize moisture pickup and facilitate handling.

**Procedure for granulation.** A granulation procedure, similar to that used in the pharmaceutical industry as described by Peek (21), was adopted. A mixer (Kenwood Chef model) was used for the first two steps. This mixer employs planetary motion, ensuring thorough mixing. The beater was used for the rewetting operation, while the colander and sieve attachment was used to force moistened particles through a screen, having a rotating paddle fitted with scraper blades which travel over the surface of a screen inside the colander bowl. In this study the finer of the two screens was employed, having holes of 1,600 μ in diameter. The speed of the mixer was regulated with a variable voltage control, as the control for the Kenwood could not be regulated to low speeds.

The four steps used in this procedure were as follows:
1). Two hundred grams of powder were placed in the mixer bowl and the beater set in motion. The quantity of water needed was allowed to drip slowly and in stages. Regular manual stirring with a rubber plate scraper was also used to eliminate quiescent spots in the mixer. Mixing required 10-15 minutes. Throughout this work, powder samples and water were used at 18 C.

2). After allowing the moistened mix to stand for 5-10 minutes, granulation was done in the colander and sieve attachment. For most of this work the moistened NFDM was granulated through a screen having holes 1,600 μ in diameter. However, granule size was also varied by varying screen size. For this purpose special screens were constructed by soldering standard wire mesh on to a perforated supporting base plate to fit into the colander attachment of the Kenwood Chef mixer.

3). The moist granules were spread on to a tray and air-dried at 60 C in a fan-assisted dryer for 50-60 minutes, drying time being adjusted to give a final product of 3.0-4.0% moisture. The granules on the drying tray were stirred periodically during drying.

4). The dried granules were passed through a 2,000-μ (B.S. 8-mesh) sieve to eliminate large clumps formed during drying.

Fig. 1. Four steps in the granulation procedure. A. Mixing and rewetting of base powder. B. Granulation in colander and sieve attachment. C. Tray drying at 60 C. D. Dry screening through 2,000-μ sieve.
The four stages in this procedure are depicted in Fig. 1.

**Results and Discussion**

*Temperature of drying of granules.* In drying moist granules it was desired to effect a minimum of heat damage. Some experimentation was done to determine a satisfactory drying temperature-time combination which would give minimum damage. The solubility index of the dried granules was the index of heat damage. Results are presented in Table 1.

**TABLE 1**

<table>
<thead>
<tr>
<th>Drying conditions on moist granules</th>
<th>Initial moisture (%)</th>
<th>Final moisture (%)</th>
<th>Solubility index (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control Niro NFDM</td>
<td>3.5</td>
<td>11.5</td>
<td>0.10</td>
</tr>
<tr>
<td>43 C/165 min</td>
<td>11.5</td>
<td>4.0</td>
<td>0.10</td>
</tr>
<tr>
<td>49 C/100 min</td>
<td>11.5</td>
<td>3.7</td>
<td>0.10</td>
</tr>
<tr>
<td>54 C/100 min</td>
<td>11.5</td>
<td>3.3</td>
<td>0.15</td>
</tr>
<tr>
<td>60 C/55 min</td>
<td>11.5</td>
<td>3.2</td>
<td>0.10</td>
</tr>
<tr>
<td>71 C/35 min</td>
<td>11.5</td>
<td>3.2</td>
<td>0.40</td>
</tr>
</tbody>
</table>

As can be seen for granules, initially at 11.5% moisture no heat damage was reflected in the solubility index until the drying temperature was raised above 60 C. It was, therefore, decided to tray-dry all granules at 60 C for 50-60 minutes, i.e., to reduce moisture to 3.0-4.0%.

*Effect of rewetting moisture.* As discussed earlier, there is considerable evidence to suggest the importance of rewetting moisture in determining stable granule or agglomerate formation. But, there is no evidence of the effect of rewetting moisture on the properties of instant powders such as instant NFDM. Employing the technique of wet granulation, it is possible to study such relationships.

Two samples of commercial NFDM, viz., Niro NFDM and Rogers NFDM, were granulated over a range of rewetting moistures. All these samples were granulated through the 1,600-μm screen and then dried and sieved as described. Rewetting moistures were determined by sampling the product after treatment in the colander and sieve attachment. Some drying occurred during granulation, as moist powder was forced through the screen, but it was not significant. It was found more satisfactory to sample after granulation.

Therefore, both powders were granulated corresponding to a range of rewetting moistures. They were analyzed for dispersibility, sinkability index, solubility index, bulk density, and porosity. Results are presented in Figs. 2 and 3.

The effect of rewetting moisture upon actual granule formation is shown in Fig. 4 for Niro NFDM.

The critical nature of rewetting moisture on the properties of the final product is self-evident in every respect. Firstly, the rewetting moisture content controlled the actual granulat-
REWETTING MOISTURE % WEIGHT

Properties of granulated nonfat dry milks (redried).

Figure 3. Properties of granulated nonfat dry milks (redried).

ing process. With increasing moisture content a point was reached where the granulated product changed from a fluffy powder with a poor flow to well-formed granules of excellent flow properties. The point at which this change occurred has been called the break point, and may be detected in Fig. 4.

It is interesting to compare the break point moistures obtained in the granulation of NFDM with those for the granulation of sand by New-

FIG. 4. Samples of redried granulated Niro nonfat dry milk showing effect of rewetting moisture upon granule formation.

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Tab and Conway-Jones (20) and Capes and Danekwerts (6). Taking the break point for NFDM at 12% moisture, the comparison is made in Table 2. A break point moisture of 12% is comparable to 16.4% on a v/v basis, and to 16% void saturation as compared with 96% void saturation for the granulation of sand. This shows clearly that void saturation is not necessary in the granulation of a noninert powder such as NFDM, adhesion of particles in this case no doubt being assisted by dissolved solids to form a binder solution.

The effect of rewetting moisture upon the reconstitution characteristics of the redried granulated powders is shown in Fig. 2. The dispersibility of both Niro NFDM and Rogers NFDM could be increased markedly, optimum conditions coinciding with break point rewetting. At this optimum, dispersibility values are equal to those of instant powders, as revealed by analysis of commercial samples by Neff and Morris (19). The sinkability index of the Niro NFDM increases with increases in rewetting moisture. At break point rewetting and above the sinkability index has been termed infinite, as the granules lose all tendency to float on the surface of the water. Sinkability index values were not determined for the Rogers NFDM, but showed an identical trend when examined qualitatively. The solubility index of samples shows a sharp increase beyond break point rewetting, whereas below break point, solubility indices are satisfactory and would pass ADMI standards of 1.0 ml.

The increase in dispersibility and sinkability index up to the break point suggests that this is due to the process of size enlargement. Decreased dispersibility beyond break point is more difficult to explain and must be attributed to increase in solubility index; that is, the decrease in solubility proper of granules produced at these higher rewetting moistures. Two further observations giving evidence of the

<table>
<thead>
<tr>
<th>Table 2</th>
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<tbody>
<tr>
<td>Comparison of break point moistures in the granulation of sand and nonfat dry milk</td>
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<tr>
<td></td>
</tr>
<tr>
<td>Density: g/cc</td>
</tr>
<tr>
<td>Void volume: cc void/g solid</td>
</tr>
<tr>
<td>Granulation break point at:</td>
</tr>
<tr>
<td>Moisture v/v</td>
</tr>
<tr>
<td>Moisture cc/g</td>
</tr>
<tr>
<td>Void saturation</td>
</tr>
</tbody>
</table>
critical nature of the rewetting moisture were made:

1) Granules produced at above break point rewetting appeared to be more yellow than samples below break point. This could be partly attributed to an optical effect, due to increased particle size (demonstrated by grinding up granules to a finer particle size), but it was concluded that some of this color change was due to a chemical effect.

2) During rewetting of NFDM, then at approximately 14% rewetting moisture, the mix passed through a definite doughy stage. This is as if a gel state had been formed during mixing.

These observations suggest that at above break point moistures, additional water is available for reaction above the minimum required to act as a binding liquid for granulation. The possibility exists that some of this water may combine with protein to form a gel; removal of such water would be more difficult upon subsequent drying and could account for the increased solubility index and decreased dispersibility. Further support for the critical nature of this rewetting moisture comes from a study of the browning reaction, with associated insolubilization, in a NFDM system. Such changes are comprehensively discussed by Coulter et al. (8). It is noted that moisture is an important factor in determining rate of browning. The optimum moisture for browning in a NFDM system is approximately 12-14%.

Samples of the redried granulated Niro NFDM were also examined for state of lactose. It was found that as rewetting moisture increased, there was a gradual increase of crystalline lactose, with a sharp change to predominantly crystalline lactose coinciding again with break point rewetting conditions.

The bulk density and porosity measurements shown in Fig. 3 are quite interesting, especially when compared with the corresponding dispersibility curves in Fig. 2. Again it is seen that minimum bulk density values, or maximum porosities, coincide with break point conditions. The strong correlation observed between dispersibilities of samples and their porosities (or inversely, bulk densities) is interesting, in view of the findings of Harper et al. (10). They consider that the concentration of milk solids in the vicinity of powder particles is an important factor in their instant solubility; hence, the lower the bulk density the higher the instant solubility of a powder. Although the effect of increasing porosity as the break point is approached would certainly assist the dispersibility of the powder, it will be shown that the effect of particle size also is most important.

Peebles (22) in his instantizing patent of NFDM notes that optimum rewetting, claimed to be 15% moisture, corresponds with a minimum bulk density of the product. He states that if too much water is introduced, it becomes readily apparent by a decrease in the apparent bulk of the material. Peebles stated this as a qualitative observation, whereas our study has shown conclusively the critical nature of rewetting moisture upon both dispersibility characteristics and bulk density of the product.

Another interesting point emerges from Figs. 2 and 3 when comparing Niro NFDM and Rogers NFDM; namely, the very narrow range of rewetting moistures over which the Rogers NFDM may be instantized. This result illustrates the notion held in the dried milk industry in the United States float some types of NFDM are comparatively difficult to instantize. The reason for this is not clear, although it was noted in our work that the sample of Rogers NFDM had an extremely low mean particle size, being less than 10 μ.

Effect of particle size. Apart from having elucidated optimum rewetting conditions, it would be desirable to determine optimum particle size of NFDM with respect to dispersibility. This was approached in three ways: 1) Granulation through different screen sizes at above break point moistures; 2) sieving of large sample produced at break point rewetting to give different size fractions; 3) grinding of large sample of granules produced at above break point rewetting, followed by sieving to obtain different size fractions.

Granulation through different screen sizes. Batches of Niro NFDM were moistened as described, using the same quantity of rewetting water in each case, so as to yield granules of approximately 14% moisture before redrying. Instead of granulation being achieved through the 1,600-μ screen, as in all the other work, in this case granulation was done through the specially manufactured screens of differing mesh size. Screens down to 80 mesh were manufactured, but the finest mesh through which moistened NFDM could be granulated was 40 mesh. Increasing difficulty was encountered in forcing the moistened mix through the screens, as the mesh aperture size was decreased. This was evident in drying which occurred during actual granulation—increasing difficulty in granulation leading to greater moisture loss. This can be seen in Table 3, showing the moisture contents of the granulated...
samples. All batches were rewet with equal quantities of water, and moistures were determined on the granulated NFDM.

Since the same amount of moisture was added to all five batches initially, these samples may be assumed to represent equivalent rewetting moistures (viz., 14.0%), the only variable being the granulation size. Good granules were formed in all cases.

Samples were analyzed for dispersibility, porosity, and solubility index. The results are presented in Fig. 5.

**Sieving of sample produced at break point.**
Four batches of Niro NFDM were granulated using the 1,600-μ screen. The same quantity of moisture was added to each batch to correspond to approximately break point rewetting. The moisture content of the moist granules was 11.6%.

Under break point conditions a wide size distribution is produced in the dried product. Production of four batches, therefore, allowed sieving to yield sufficient fractions in each size range for analysis. Eight sieve fractions were obtained and analyzed for dispersibility and sinkability index, the results being presented in Fig. 6. In addition, a particle size distribution curve was obtained and is presented in Fig. 7.

**Sieving of ground granules.** Four batches of Niro NFDM were granulated, using the 1,600-μ screen. Again equal quantities of water were added to each batch but this time corresponding to 14.0% moisture in the moist granules. The dried granules were then ground to give a wide size distribution for subsequent sieving. It was found that an attrition mill, or coffee grinder, gave a wide size distribution suitable for this purpose.

The ground granules were sieve-separated to give eight fractions, which were analyzed for dispersibility and sinkability indices. The results are presented in Fig. 6.

From Fig. 5 it can be seen that dispersibility of a redried granulated product increases as the granulation size is decreased from 2,000 to 390 μ. It must be remembered that these granules were produced by rewetting to 14.0% moisture, which is well beyond the optimum for reconstitution. Yet, even so, the dispersibility is increased from 25.0 to 54.3% merely by decreasing granulation size. However, as wet granulation could not be achieved through finer screens, an optimum size for granulation has

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**TABLE 3**

Moisture content of nonfat dry milk granulated through different size screens

<table>
<thead>
<tr>
<th>Screen size</th>
<th>Aperture (μ)</th>
<th>Moisture (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 Mesh</td>
<td>2,000</td>
<td>14.0</td>
</tr>
<tr>
<td>Kenwood screen</td>
<td>1,600</td>
<td>13.6</td>
</tr>
<tr>
<td>16 Mesh</td>
<td>1,000</td>
<td>13.7</td>
</tr>
<tr>
<td>30 Mesh</td>
<td>500</td>
<td>13.3</td>
</tr>
<tr>
<td>40 Mesh</td>
<td>390</td>
<td>12.7</td>
</tr>
</tbody>
</table>

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**Fig. 5.** Effect of granulation screen size on properties of nonfat dry milk.

**Fig. 6.** Effect of particle size on reconstitution of nonfat dry milk.
not been determined. All that can be deduced from Fig. 5 is that an optimum granulation size will be less than 390 μ.

Porosity and solubility index determinations in Fig. 5 show only very slight change with granulation size. This is as would be expected as, theoretically, porosity will be independent of particle size, providing a population consists of spherical particles all of the same diameter.

Figure 6 shows the effect of particle size upon reconstitution characteristics for a more complete particle size range. In this instance, an optimum particle size has been determined. In both granulated powders, that is, rewetting to 11.6 and 14.0% moisture, the optimum particle size has been determined at approximately 200-μ diameter. This optimum represents optimum dispersibility togerher with infinite sinkability. It may be emphasized that the maximum dispersibility of 75% obtained for the optimum size, optimum rewetting fraction is higher than any commercial instant powders tested by Neff and Morris (19).

It should be noted that beyond 130 and 180 μ particle size for the 14.0 and 11.6% rewet granules, respectively, there was an infinite sinkability index, when the powder no longer floated on surface of the reconstituted liquid. This phenomenon of a particle sinking beyond a critical size would require a knowledge of solid-liquid surface tensions and contact angles at the water surface for any quantitative treatment. However, qualitatively this change to infinite sinkability may be explained as follows: The force tending to submerge a particle on the surface is its weight less the buoyancy effect. As the radius, R, of a particle increases, this force will increase as a function of R². On the other hand, the force supporting the particle on the surface is due to a surface tension effect, being a function of perimeter in contact with the water. This supporting force will vary with R only. Therefore, on increase in particle size the surface perimeter-to-mass ratio of the particle will decrease. It is reasonable to predict, therefore, that a critical size exists where the force tending to submerge the particle becomes greater than the force tending to support the particle at the surface. This critical size appears to be approximately 130-180 μ for NFDM granules.

From the particle size distribution curve in Fig. 7 it is seen that at break point rewetting the final product consists of a population with two definite peaks at 130 and 680 μ. This is interesting, because optimum size is approximately 200 μ, indicating that dispersibility
of this product could be increased further by reducing the oversize fraction.

Since the weight fraction and dispersibility of each size fraction is known (Fig. 6 and 7) in the case of the 11.6% rewet granulation, it is possible to calculate a weighted average dispersibility for the mixed granulation. The weighted average dispersibility is 52.7%. In comparison, the measured dispersibility of a sample of the nonfractionated granulation is 52.4%. This close agreement emphasizes the importance of particle size in influencing dispersibility.

Figure 6 serves well to illustrate the importance of both particle size and rewetting moisture on the dispersibility of the final redried granulated product. The 14.0% rewet granulation exhibits a lower dispersibility than the 11.6% rewet product at any particular particle size. For example, at 400 μ size the former product has a dispersibility of 25.3%, while the 11.6% rewet product has a dispersibility of 67%.

Acknowledgements

The authors gratefully acknowledge the advice of and discussion with G. J. S. Latimer of the Food Technology Department, Massey University, and the cooperation of various staff members of the New Zealand Dairy Research Institute.

References