RECONSTITUTION CHARACTERISTICS OF FOOD POWDERS AND GRANULES WITH EMPHASIS ON NON FAT DRIED MILK

A Thesis presented in partial fulfilment of the requirements for the Degree of Master of Food Technology in Food Processing at Massey University, Palmerston North, N.Z.

Edward Neff

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"When you know a thing, to hold
that you know it; and when you
do not know a thing, to allow
that you do not know it;
this is knowledge".

CONFUCIUS

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ABSTRACT

Reconstitution characteristics of food powders form a major determinant of consumer acceptance, particularly with the trend to instant foods. A working theory has been proposed to explain the mechanism of dispersion of any soluble food powder.

A study was made of methods which may be employed to modify reconstitution characteristics. No significant improvement in the dispersibility of Non Fat Dried Milk (NFDM) could be achieved by a compression/repowdering process even though the particle density and porosity of the powder could be increased by this technique. When applied to spray dried instant coffee such process of compression, up to 150 psi, resulted in a small improvement in dispersibility while at the same time achieving a marked increase in the bulk density of the powder. The significance of this observation with regard to potential saving in packaging volume has been discussed.

The most significant improvements in reconstitution characteristics of NFDM were achieved by a rewetting/redrying process. A granulation technique is described which has been successfully employed to simulate commercial instantising of powders. By means of this granulation technique it has been shown that by far the most important factors in agglomeration influencing the properties of the resultant "granules" are :

1. Rewetting moisture content at which granulation is achieved prior to redrying;

2. Particle or granule size of the final product.

Optimum conditions for NFDM have been determined to be 11-12% rewetting moisture and a mean particle size of 200/u. This granulation technique has also been employed to study the effect of additives at agglomeration upon reconstitution properties of NFDM.

Several commercial processes are in use, and are covered in patents, for the purpose of instantising NFDM and other food powders. Despite this, however, no study has previously demonstrated the critical nature of certain variables in this process as clearly as has been done in this study.

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I. INTRODUCTION

It cannot be disputed that the trend today is towards instant and convenience foods. At the same time there has been considerable expansion in the spray drying of food powders; in particular, in the dairying industry the last few years has seen the installation of a large number of high capacity spray drying plants both in Australia and New Zealand. One of the problems, however, is that many of these conventionally spray dried powders among them Non Fat Dried Milk (NFDM) possess poor reconstitution or dispersibility characteristics. This has prevented widespread acceptance of such powders as a household item and has restricted the development of new markets. As an example it may be pointed out that by far the major portion of the market for milk powders still relies on industrial recombining and reconstitution facilities and sale of the liquid products. This obvious market prejudice against powders must be, at least in part, due to poor dispersibility characteristics. It is the aim of this study to examine more closely factors associated with dispersibility or reconstitution properties of powders.

Taking again spray dried NFDM as an example it must be emphasised that this possesses excellent ultimate solubility. For example, A.D.M.I. standards for Extra Grade NFDM require a solubility index of not greater than 1.25 ml. This is equivalent to a solubility of greater than 96%. The problem, therefore, lies not with ultimate solubility but with its instant solubility or dispersibility, as would be encountered by a consumer attempting to

reconstitute NFDM with water. Admittedly, the dispersibility of NFDM is not the prime consideration when referring to reconstitution on a large scale as is the case in commercial recombining plants in operation in South East Asia. But, dispersibility of a food powder still remains a major quality attribute where reconstitution is to be performed by the consumer immediately before use. In view of the trend towards instant foods, therefore, a better understanding of dispersion of food powders is paramount to technological progress in this field.

Evidence suggests that the dispersibility characteristics of a powder are associated mainly with physical properties. This is suggested by the fact that a conventionally spray dried NFDM of poor dispersibility can be converted to an "instant" powder by an agglomeration process involving rewetting and redrying to produce a powder of increased particle size.

Research on reconstitution characteristics of food powders has been examined in three sections :

(1) Characterisation of Typical Food Powders

This includes the development of test methods to allow characterisation of powders culminating in the postulation of a working theory of the mechanism of dispersion of any food powder.

(2) Effect of Non-Rewetting Processes Upon Dispersibility

This examines possible techniques which may be employed to modify reconstitution characteristics without requiring a rewetting operation.

(3) Effect of Rewetting Processes Upon Dispersibility

This examines a rewetting process, as in commercial instantising, employed to improve the dispersibility of NFDM.

II. METHODS OF ANALYSIS

A. LITERATURE REVIEW

Analytical techniques were required for the determination of dispersibility charactersitics of a powder and also physical properties such as density, particle size, etc. Comparatively little difficulty was encountered in the selection of techniques for the measurement of physical properties. However, the evaluation of dispersibility characteristics proved to be a more elusive task.

The difficulty in designing an objective test of dispersibility, or ease of reconstitution, is best illustrated by reference to a review by King (1966), wherein scores of tests are described which have been employed for such evaluation of milk powders. King (1966) categorises these tests as :

(a) Determination of wettability;

(b) Determination of self-dispersion and of dispersion at low energy stirring;

(c) Determination of sinkability.

These divisions seem rather artificial since sinkability will be influenced by wettability, as will self-dispersion. It was, therefore, decided to examine the tests in perspective to consumer requirements for a powder. It was soon appreciated that no one test

would fully evaluate ease of reconstitution as judged by a consumer. Resigned to the fact that at least two test methods would be required it was decided that the two most important factors in evaluating ease of dispersion are :

(i) Sinkability

i.e. disappearance of powder from an unagitated water surface;

(ii) Dispersibility

i.e. ability of powder to disperse when mixed with water in a manner comparable to reconstitution by consumer.

In searching for a suitable sinkability test a technique was desired which would give a measure of the ability of a powder to disappear from a quiescent water surface. Reports of such tests include Kleinert (1950); Ashworth and Gunthardt (1954); Mather and Hollender (1955); Baker and Bertok (1959); Bullock and Winder (1960); and Radema and van Dijk (1962). These workers all describe tests involving the spreading of varying amounts of milk powder on the surface of water and measuring the solids passing into "solution" at varying intervals either by direct sampling and solids determination or by photometric measurement. Muers and House (1962) describe a method of spreading a sample of milk powder on a quiescent water surface and measuring the time for complete wetting. Mol and de Vries (1962) describe a technique for continually feeding a stream of milk powder onto a stream of water and measuring the reflection from the surface of a light beam to give an index of wettability.

Many of these tests appeared cumbersome and the major

problem appeared to be in achieving a uniform distribution of powder onto a quiescent water surface. For this reason a sinkability test was developed for the purpose of this research.

The problem of designing a suitable dispersibility test to evaluate the ability of a powder to redisperse to a fluid milk has been well discussed by Stone (1955). He describes the development of a dispersibility test wherein 52gm of powder are mixed with 400ml of water in a Hobart kitchen mixer. In this instance, rotation of the agitator was controlled at 135 rpm and continued for varying periods of 10-40 seconds. Upon completion of stirring, the reconstituted milk was drained from the bottom of the mixing bowl and passed through a 210/u screen before determination of total solids of an aliquot.

Moats et al (1959) modified this test by varying the speed of rotation and reducing mixing time to 5 seconds, claiming that this gave better sensitivity. They also employed a hydrometric method to determine the solids dispersal.

The American Dry Milk Institute (A.D.M.I.) adapted the dispersibility method of Stone et al (1954), to the testing of instant Non Fat Dried Milk (NFDM). A.D.M.I. specify the mixing of 52gm of powder with 400ml of water at 75°F for a period of 20 seconds, using a speed of rotation of 192 rpm. It was decided to adapt this test for dispersibility evaluation in this research. Since a Hobart kitchen mixer was not available, some modifications were necessary to the A.D.M.I. procedure.

B. PROCEDURES

(i) Sinkability Index (Original Method)

As mentioned previously, the major problem with all reported methods (variously called sinkability, self-dispersion, or wettability) involved the placing of a sample of powder uniformly onto a quiescent water surface. The procedure here adopted may best be described by reference to Figure 1.

A sintered glass funnel is rigidly mounted on a stand and a disc of filter paper is cut to 6cm diameter (28.2cm^2 area), so as to just fit on the sintered glass plate of the funnel. A known weight of powder is then placed onto the filter paper and carefully spread to a uniform layer by means of a fine brush. The filter paper is next held down onto the sintered plate by means of two wire clips. Water, at 75°F, from a thermostatically controlled elevated tank is then carefully allowed through the bottom of the funnel by opening the pinch clamp on a connecting rubber tubing. This allows the powder sample to be floated on the surface of the water without causing any disturbance of the surface, while maintaining a uniform distribution of powder on the surface. The time required for all the powder to disappear from the surface is measured; whether this be by self-dispersion or sinking without dispersion.

This technique, therefore, makes possible the determination of the time required for a weight of powder to disappear from a water surface. This information alone is not sufficient to compare different powders since there will not be a linear relationship

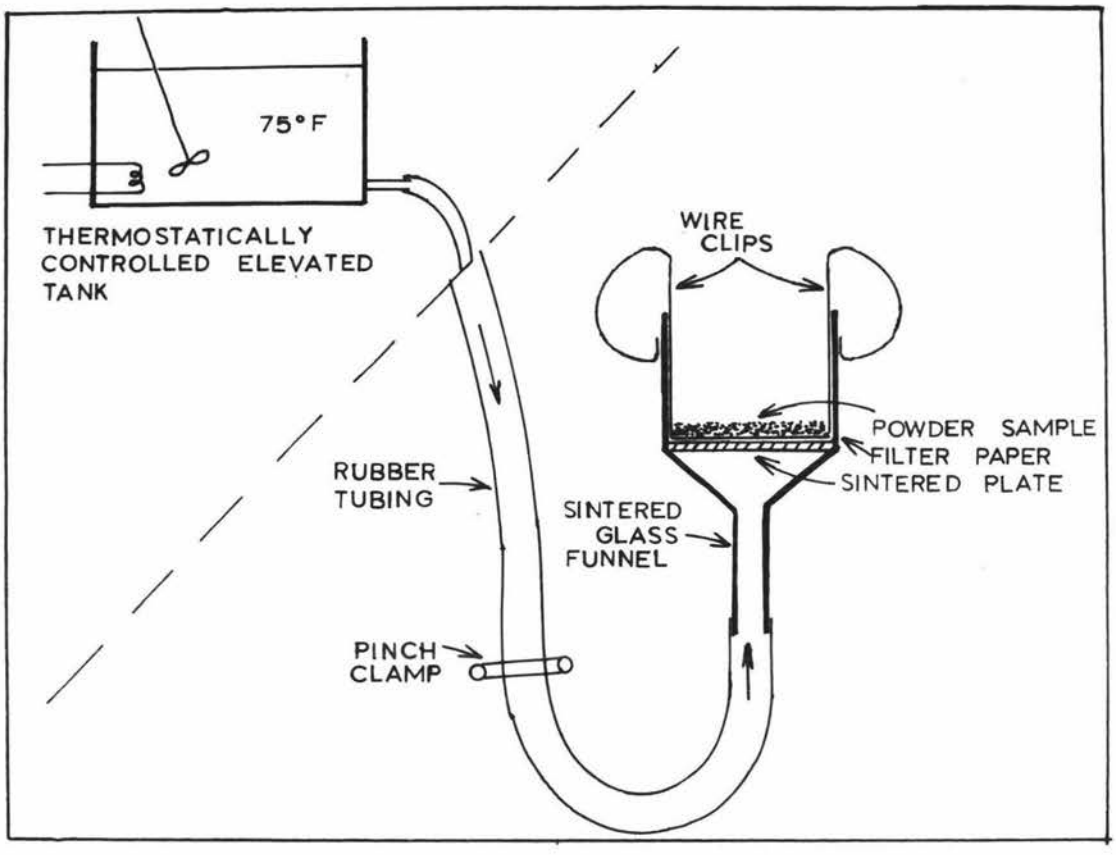

FIG.1 APPARATUS FOR SINKABILITY INDEX DETERMINATION

between the loading of the water surface and time for complete disappearance. Therefore, times for sinking are measured for different weights of powder so as to allow the plotting of results as in Figure 2. Then, in order to compare different powders the weight sinking in 20 minutes is interpolated from the graph and the sinkability index is expressed as mg of powder sinking per minute per square cm of area, (i.e. mg/min.cm^2).

Typical plots obtained for several powders are shown in Figure 2. It may be noted that this method is applicable to any food powder, not necessarily milk powder.

(ii) Dispersibility (Modification of A.D.M.I. Method)

It was decided to adapt the A.D.M.I. technique for the determination of dispersibility of Instant NFDM as closely as possible using apparatus available locally.

Since a Hobart mixer was not available, the mixer chosen was a Kenwood "Chefette". Tests were first carried out with different shaped bowls and beakers, different mixing speeds and different mixing times. The A.D.M.I. method requires the mixer to be hinged so as to allow the beaters to be lowered into the bowl. It was first thought that the Kenwood mixer could be manually raised and lowered vertically on a laboratory stand. Another factor concerned the use of one or two beaters in mixing. The A.D.M.I. method employs one large whisker-type beater. However, the Kenwood mixer possesses two smaller beaters which may be used singly or

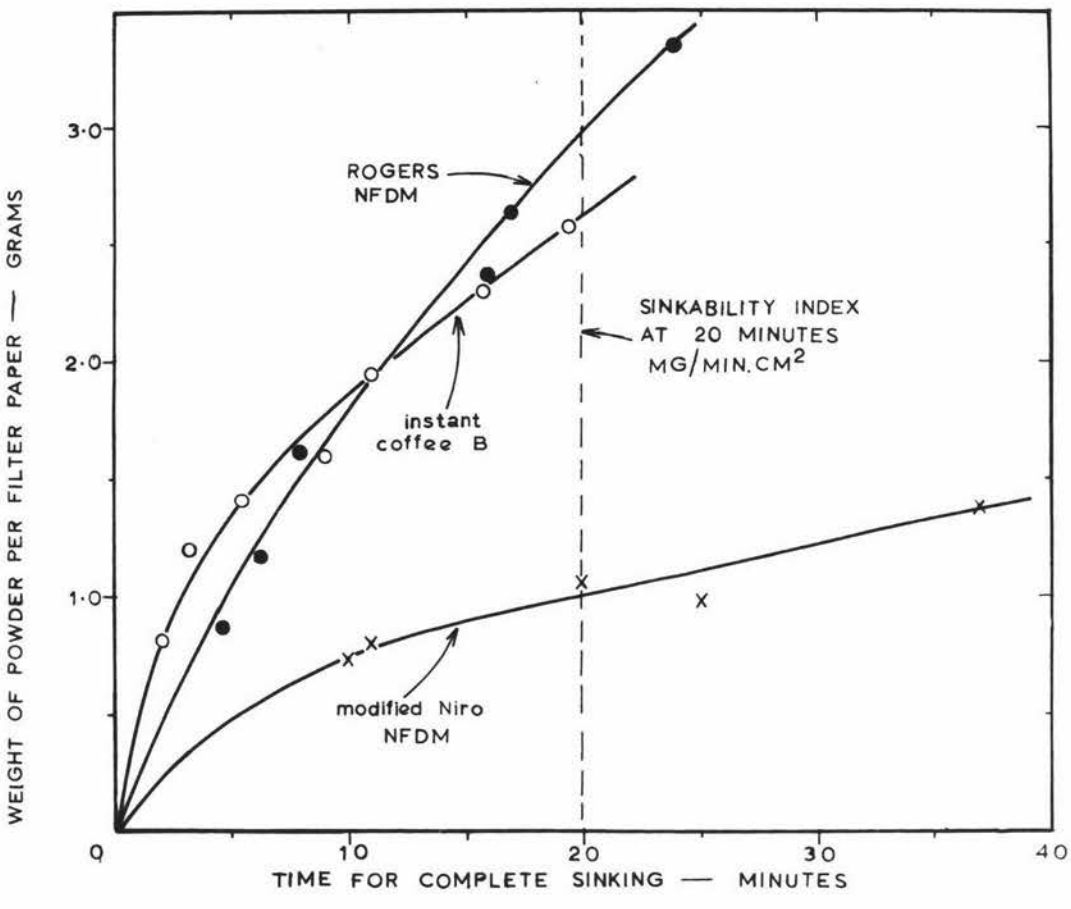

FIG. 2 TYPICAL SINKABILITY CURVES

together. These factors had to be determined and reproducibility tests were carried out as each technique was improved. Reproducibility was determined by carrying out 10 dispersibility determinations on the same sample of Niro NFDM. These results are best presented in tabular form in Table 1 :

TABLE 1

Reproducibility of Dispersibility Test

Details of Procedure	No.of Tests	Mean Value	Standard Deviation
1. Using 1 beater, manually raising and lowering mixer vertically, mixing 350 rpm/ 20 seconds.	10	41.1%	11.3
2. Using 2 beaters, mixer hinged for raising and lowering, mixing 175 rpm/20 seconds.	10	32.5%	1.9
3. Using 2 beaters,etc. as for 2, bowl more rigidly fixed and operation streamlined.	10	35.0%	1.8

As can be seen the variation in the test was eventually reduced to a standard deviation of 1.8 which was considered satisfactory. The procedure employed may be described by reference to Figure 3.

The Kenwood mixer is clamped in position using a laboratory clamp and stand in such a way that it can be raised and lowered about a fulcrum as shown. A stop is provided to allow the mixer to be lowered to the same position consistently. Two beaters are fitted to the mixer and these are adjusted to rotate at 175 rpm by means of

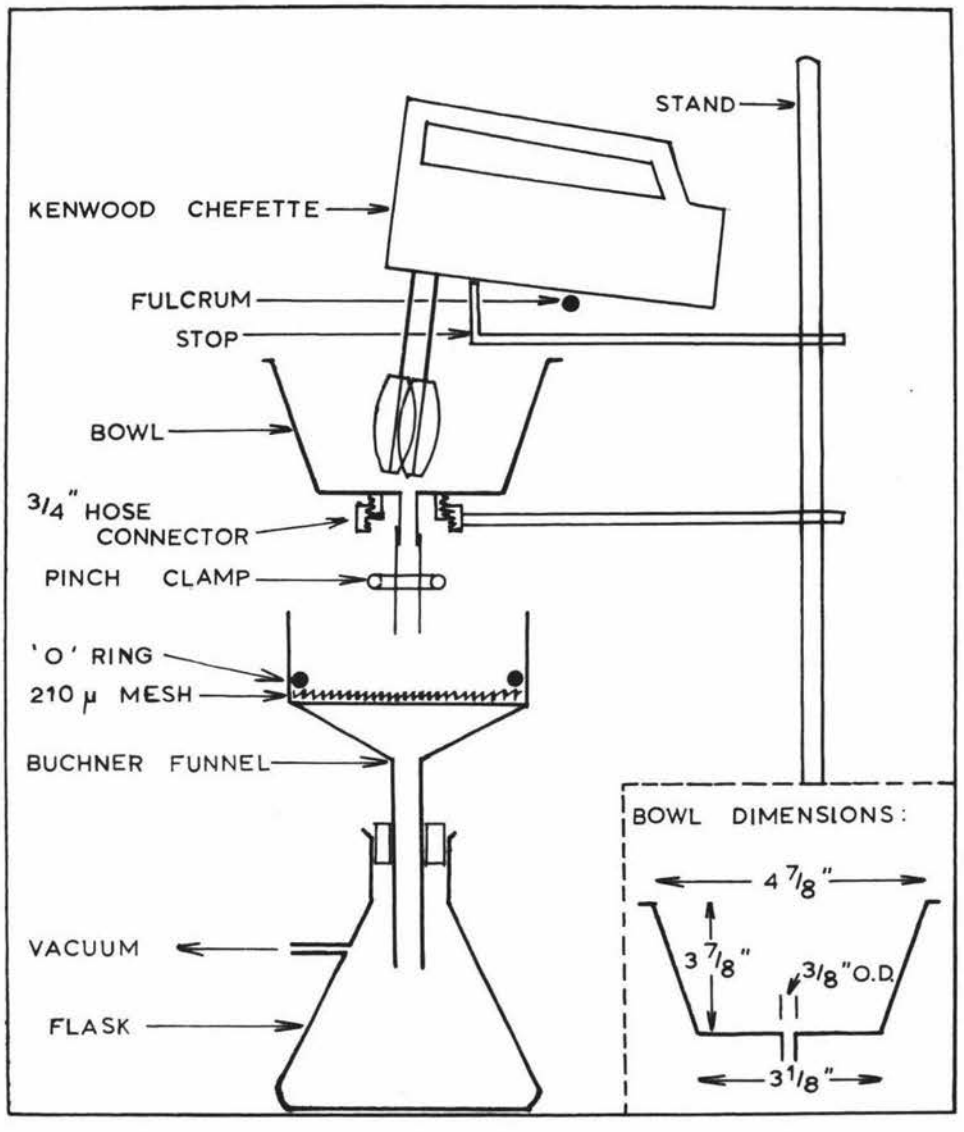

FIG. 3 APPARATUS FOR DISPERSIBILITY TEST

a "variac" variable voltage control, in the power supply to the mixer. Adjustment is made by means of a tachometer and it was found necessary to adjust the setting immediately before each test. The mixing bowl* is placed in position and can easily be removed and replaced to the same position by means of a $\frac{3}{4}$" hose connector assembly, one part of which is brazed to the bowl, the other being held in position by a connecting arm to stand. The bowl is fitted with a $\frac{3}{8}$" O.D. outlet tube which can be closed off by means of a pinch clamp over rubber tubing. Below the bowl is arranged a Buchner funnel and flask as shown. A disc of 210/u screen (70 mesh) is held in the funnel by means of a rubber "O" ring.

In the testing of powder samples a procedure similar to that of A.D.M.I. is followed. Three hundred ml of water at 75°F is placed into the bowl and 39gm of powder is transferred to the surface of the water. (This is in the same proportions to the A.D.M.I. method where 400ml and 52gm, respectively, are employed). The mixer is then turned on, rotation being 175 rpm, and lowered into the bowl. Agitation is continued for 20 seconds, after which the beaters are left in their position in the bowl. The pinch clamp on the outlet of the bowl is then opened to release the contents onto the 70 mesh screen. The screened liquid is collected in the filter flask and diluted to 500ml. Two 10ml portions are then transferred to two weighed aluminium dishes, evaporated to dryness on a steam bath and dried at 102°C/3 hours. This allows the weight of solids dispersed to be calculated. This is expressed as percent dispersibility.

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* A 'Dissco", size 5, stainless steel bowl was employed, manufactured by Dunedin Stainless Steel Co.Ltd, 33 Creswell Street, Dunedin, N.Z.

It was found, after testing many samples, that this method is in fact more sensitive than the A.D.M.I. method. Reports in the literature, Tamsma et al (1965), Hanrahan and Kontson (1965) suggest that the A.D.M.I. method continually gives a dispersibility figure of above 90% for instant milk powders. This makes it very difficult to attach any significance to changes in dispersibility between 90 and 100 percent. This modified method, however, would appear to be more sensitive in this respect since the highest dispersibility figure obtained with a commercial instant NFDM was 69%.

(iii) Solubility Index (Standard Method)

Although many objections have been raised at the A.D.M.I. method of solubility index determination, Steen (1962); van Kreveld and Verhoog (1963), it still remains the most widely accepted method today. Therefore, it seemed reasonable to follow this method as closely as possible.

The only variation necessary to the A.D.M.I. method was with regard to the special mixer which was not available locally. This was overcome by employing the liquidiser attachment together with a Kenwood "Chefette" mixer. Steen (1962) describes the A.D.M.I. mixer as having an impeller speed of 3,400 rpm. The Kenwood mixer was adjusted to this speed by means of a "variac", variable voltage control, in the power supply to the motor. Due to the dimensions of the liquidiser attachment, it was more convenient to mix 20gm of NFDM (or 26gm of Full Cream Dried Milk (FCDM)) with 200ml of water. The A.D.M.I. method specifies 10gm of NFDM (or 13gm of FCDM) with

100ml of water.

In all other respects the A.D.M.I. method was adhered to. The solubility index is expressed as ml of sediment.

(iv) Moisture Content (Standard Methods)

For the section of experimental work dealing with the characterisation of food powders, moisture determinations were carried out by the Toluene Distillation method as specified by A.D.M.I. Care was taken to extend the distillation time until the reading in the moisture trap remained constant. This precaution will be appreciated by reference to Choi et al (1948) who show that water of crystallisation requires an extended distillation time if it is to be desorbed.

For the section of work dealing with rewetting processes, an oven drying moisture determination was employed. Weighed samples were dried in a fan assisted air oven at 102-103°C/3 hours.

All moisture contents are expressed on a "wet weight" basis.

(v) Bulk Density, Particle Density, Porosity

King (1954),(1965) reviews fully the physical structure of dried milk, including references to methods of determination.

Briefly, particle density of a powder will be a finite value representing the density of particles excluding voids between particles. Bulk density and porosity on the other hand, will be a function of packing of the powder mass. It is common, therefore, to

evaluate bulk density as :

1. Tapped Bulk Density

   i.e. powder mass is subjected to a standard compaction treatment;

2. Loose Bulk Density

   i.e. powder mass not compacted.

Tapped bulk density was determined by weighing approximately 70ml of powder into a 100ml measuring cylinder and giving this five sharp taps before reading the volume of powder. Tapped bulk density is expressed as gm per cc.

Particle density, loose bulk density and porosity (loose), were determined by the method of Beckett et al (1962). This method is a liquid displacement method employing hexane as a low specific gravity, inert liquid. The method is easily applied to NFDM and powders other than NFDM providing their particle density is above 0.8gm/cc. In the case of less dense powders the method could still be employed for determining particle density by use of a wire gauze disc soldered to a piece of wire which could be used to submerge all particles in the hexane. It should be noted that Beckett et al (1962) do not give details of porosity evaluation. However, it is soon realised that porosity can be obtained from this method since :

$$\text{Percentage Porosity} = 100 - \text{Percentage of Bulk Volume Occupied by Powder Particles}$$

Porosity is expressed as cc of void per cc of bulk powder (loose).

(vi) Particle Size

Methods which may be used for particle size measurement are well discussed by Irani and Clayton (1963). For spray dried powders such as NFDM sieving techniques present difficulties; other alternatives being sedimentation techniques and microscopy.

The method finally adopted is based on a microscopic technique as described by Janzen et al (1953). 0.2gm of powder is mixed with 5ml of paraffin oil and a drop of this is placed on a microscope slide and covered with a cover slip. Measurement is made with an ocular disc calibrated against a stage micrometer and 100-200 particles are measured. A modification found successful was to use a projection microscope; measurement being made on the glass luminescent scope which had been calibrated against a stage micrometer. This technique greatly eased the tedium of particle size measurement.

In the measurement of spray dried coffee particles it was found that suspension in oil and covering of a slide with a cover slip caused fragmentation of particles. This would lead to erroneous results for measurement of particle size. In this case measurement was made by sprinkling a small sample of powder onto a slide and measuring particles before too much moisture sorption had occurred.

The expression and calculation of results is best illustrated by reference to Figure 4, showing typical results for several powders. The results of microscopic measurement are grouped into the various size ranges observed. The percentage of particles falling into each size range are then expressed on a

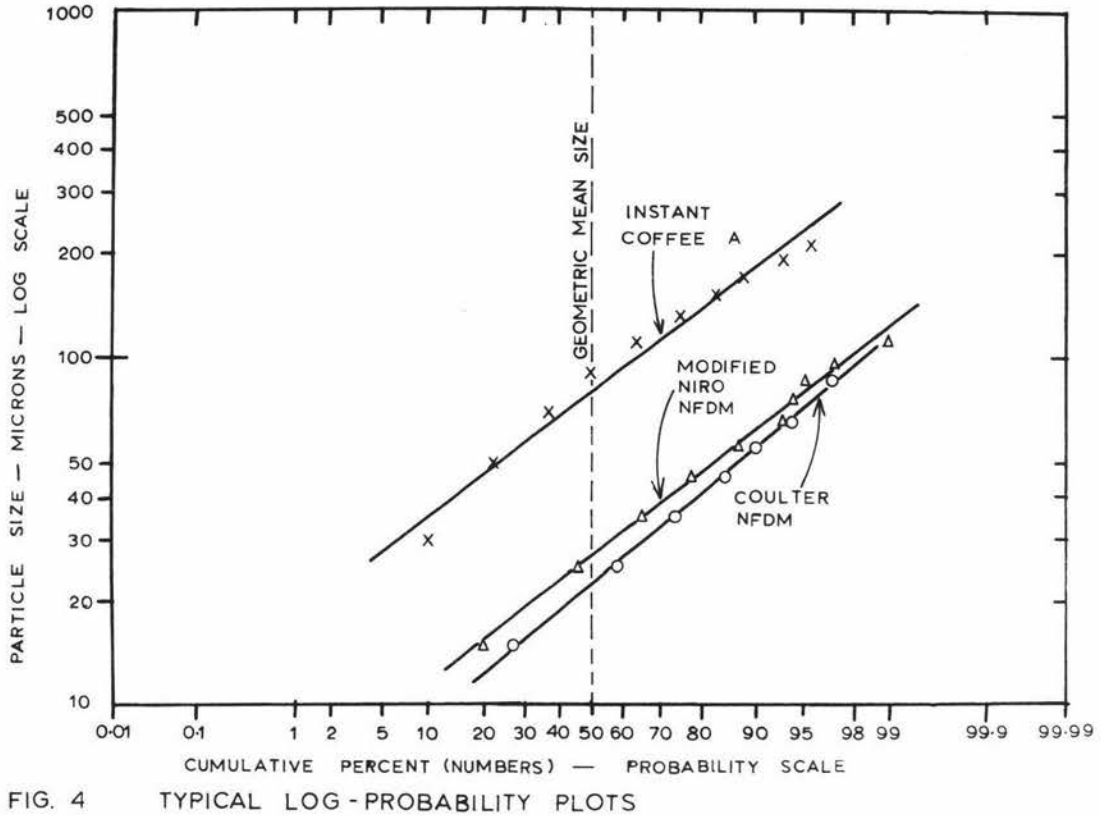


FIG. 4 TYPICAL LOG-PROBABILITY PLOTS FOR PARTICLE SIZE ANALYSIS

cumulative basis with increasing size of particles. This allows plotting of results as shown in Figure 4, on log/probability paper.

All samples examined approximated closely a straight line relationship on such a log/probability plot. Under such conditions the following population characteristics can be defined :

1. Geometric mean particle size (based on numbers) is represented by the interpolation of size at 50% cumulative;

2. Geometric Standard Deviation (Based on numbers) $= \frac{\text{Size at 85\% Cumulative}}{\text{Size at 50\% Cumulative}}$

This method of presentation and calculation of population characteristics is described by Zenz and Othmer (1960).

(vii) Photomicrographs

Photomicrographs were obtained by use of a Leitz Photomicrograph Unit. Samples were mounted on a microscope slide as described in Particle Size Analysis.

(viii) Determination of Crystalline Lactose

The state of lactose in a powder can be readily determined by means of polarised light microscopy as described by King (1948). Upon total extinction of light, using a polariser attachment, lactose crystals are readily identified as they reflect light.

III. EXPERIMENTAL WORK

SECTION (1) CHARACTERISATION OF TYPICAL FOOD POWDERS

III. EXPERIMENTAL WORK

SECTION (1) - CHARACTERISATION OF TYPICAL FOOD POWDERS

A. INTRODUCTION

A large range of food powders is to be found on the market today. Many of these are readily accepted as "instant" foods while others, such as conventionally spray dried NFDM, are more difficult to disperse on reconstitution. Further, there are several different types of spray driers employed in the manufacture of NFDM resulting, to some extent, in different powder characteristics. Although isolated references are to be found in the literature of different powder characteristics it is impossible to assess these in any comparative manner. It appeared justifiable, therefore, to analyse a range of typical food powders, including different NFDM powders, for dispersibility characteristics and physical properties.

The purpose of such a comparative evaluation is twofold :

1. It allows a comparison in perspective of the range of dispersibilities and physical properties encountered in such powders;
2. It allows a better insight to be gained into the mechanism and physical properties involved in dispersion of a powder, thus allowing postulation of a working theory.

Characterisation of food powders led to research work which may be described under three headings, viz. :

(i) Moisture determination by azeotropic distillation;

(ii) State of lactose in commercial NFDM;

(iii) Working theory of mechanism of dispersion.

B. LITERATURE REVIEW

(i) Moisture Determination by Azeotropic Distillation

Kumetat (1955) discusses the various methods of moisture analysis employed for milk powders. These include an oven method at 102-103°C, a vacuum oven method, a toluene distillation, and a Karl-Fischer titration. It is pointed out that the Karl-Fischer titration method determines all water present including the water of crystallisation of the lactose ∝ hydrate. In NFDM if all lactose were as ∝ hydrate then the Karl-Fischer method would give a reading of 2.5% higher than, say, an oven method. Kumetat states that this fact would only be of significance in the moisture determination of instant powders containing crystalline lactose.

Choi et al (1948) discuss the effect of the presence of crystalline lactose ∝ hydrate upon a toluene distillation. They describe a moisture desorption method capable of estimating the water of crystallisation of ∝ hydrate using a toluene distillation. The method depends on the difference in rates of dehydration of the crystalline ∝ hydrate from other moisture adsorbing constituents. They claim that the rate of dehydration of crystalline ∝ lactose hydrate follows a first order kinetic expression. This means that if the dehydration of a powder sample during toluene distillation is plotted on semi-log paper then dehydration of any ∝ lactose hydrate

present will result in a straight line relationship.

The A.D.M.I. specifies a toluene distillation for moisture determination. The method emphasises, without qualification, that "for some products a 60 minute distillation period is not sufficient". As toluene distillations were employed in the characterisation of typical powders it appeared worthwhile to study the rates at which water was desorbed and to note the effect of crystalline ∝ lactose upon dehydration rates.

(ii) State of Lactose in Commercial NFDM

It is generally accepted that in commercially spray dried and roller dried milk powder the lactose occurs in an amorphous glassy state. This was probably first investigated by Troy and Sharp (1930). Since then many researchers have again confirmed the occurrence of lactose in powders in a glassy state. Such reports are discussed in reviews by King (1954),(1965). It is thought that this glassy lactose constitutes a continuous phase, or hollow shell, in spray dried powder.

It is appreciated that if milk powder is permitted to take up moisture from the atmosphere then lactose crystallisation will occur; Troy and Sharp (1930); Bushill et al (1965). It is also realised that lactose will occur in a crystalline state to a variable extent in some forms of instant NFDM; Bergsoe (1957). King (1954), (1965) reports a paper by Mohr and Koenen (1951) stating that crystalline lactose had been observed in freshly made powder. In this particular instance it is inferred that this is as a result of

faulty manufacture. Apart from this isolated report in 1951 it has always been accepted that lactose in milk powder occurs as an amorphous glass.

(iii) Working Theory of Mechanism of Dispersion

Pyne (1961) has undoubtedly made the most significant contributions towards an understanding of the mechanism of dispersion of food powders, even though he studied milk powders only. Pyne states : "A basic physical concept of the process of reconstitution has never been established although many studies of the reconstitution phenomena have been made".

Pyne's hypothesis is that reconstitution is governed by capillary movement of the reconstituting liquid into the interspaces of the powder mass. Capillary movement of water into the mass allows separation of particles from the mass and hence allows easy reconstitution of those particles.

In order to prove this hypothesis, Pyne first showed that the interspaces of milk powders could act as capillaries in a predictable manner when employing non-solvating liquids. The equilibrium height of rise of a liquid in packed columns of powder was shown to be a function of pore size and could be predicted by a constant, 0.232, times the average particle radius. Further, the capillary properties of a powder could be represented by a variable termed the hydraulic radius. The hydraulic radius relates the surface contacted by liquid to the volume of liquid associated with it. The larger the particle size, the larger the hydraulic radius,

and the more rapid is the movement of liquid in the capillary. Pyne showed that there exists a linear relationship between the square of the hydraulic radius and the rate of reconstitution of a powder.

Pyne admits that his hypothesis cannot account for powders having a particle density less than 1gm/cc since in this case a particle does not separate from the powder mass since it is less dense than water. Reconstitution must occur at the powder/water interface.

Other workers have attempted to elucidate the mechanism of dispersion, or to explain factors responsible for the greater dispersibility of instant milk powders as compared with original base powder. No other work would appear to be as conclusive as that of Pyne (1961).

Bockian et al (1957) attempted to explain the greater dispersibility of instant NFDM. They found that instantised powders contained crystalline lactose although this appeared to have little significance. They found little difference in the protein nitrogen distribution between all dried milks studied. There was a large difference in size between instant and ordinary NFDM. This was considered to have a significant effect on dispersibility. Using a successive washing technique these workers were able to show that in instantising of NFDM there occurred a re-orientation of components with the highly soluble components such as salts and lactose being concentrated towards the surface. This factor was also considered to contribute towards the higher dispersibility of NFDM.

Holsinger et al (1964) modified the washing technique of

Bockian et al (1957) and also employed the technique to study orientation of components in powders made by different drying techniques. Their results were not in complete agreement with Bockian et al (1957) but they did show that instantising tended to increase the concentration of osmotically active material at the surface.

Harper et al (1963) studied the instant solubility of milk powder particles from the point of view of concentration of milk solids in the vicinity of the powder particle. The lower the bulk density of a powder, the higher the porosity, and hence the lower will be the milk solids concentration in the vicinity of each particle during reconstitution. This effect was studied by dispersing milk powders in varying amounts of sand and measuring instant solubility by a washing technique. It was shown that as the proportion of sand was increased, that is, the effective bulk density is decreased, then the instant solubility increased. The authors concluded that the bulk density of a powder must be one of the quality limiting factors in producing a self-dispersing milk powder. They set an upper limit of 0.4gm/cc for the bulk density of a self-dispersing dried milk.

Mori and Hedrick (1965) studied the effect of processing factors during instantising upon dispersibility and bulk density of the resultant powder. The most significant change of dispersibility occurred with increasing fat content in the powder.

Tamsma et al (1965) studied a range of NFDMs produced by different drying techniques for their dispersibility, sinkability, bulk

density and particle density. Some general trends are suggested but no firm conclusions are made.

From such a literature review it appeared desirable to develop a more general working theory of the mechanism of dispersion of any food powder.

C. RESULTS AND DISCUSSION

(i) Moisture Determination by Azeotropic Distillation

Powder samples were analysed for moisture content by means of toluene distillations. Rates of water desorption were noted by reading the quantity of water collected in the distillation trap at regular intervals. The distillations were continued at a uniform rate until no further water was desorbed. Results were expressed as percent moisture remaining in powder at any particular time and these were plotted on semi-log paper. The graphs obtained for several powders are shown in Figure 5.

Nearly all samples behaved in a manner similar to the "Gray-Jensen NFDM". (This represents NFDM spray dried in a Gray-Jensen drier). That is, nearly all samples showed no further water desorption after approximately 40 minutes of distillation time. However, two samples in particular, the spray dried tomato powder and the Modified Niro NFDM showed much slower rates of moisture desorption (Figure 5).

Microscopic examination, using polarised light, revealed that the Modified Niro NFDM contained crystalline lactose while the

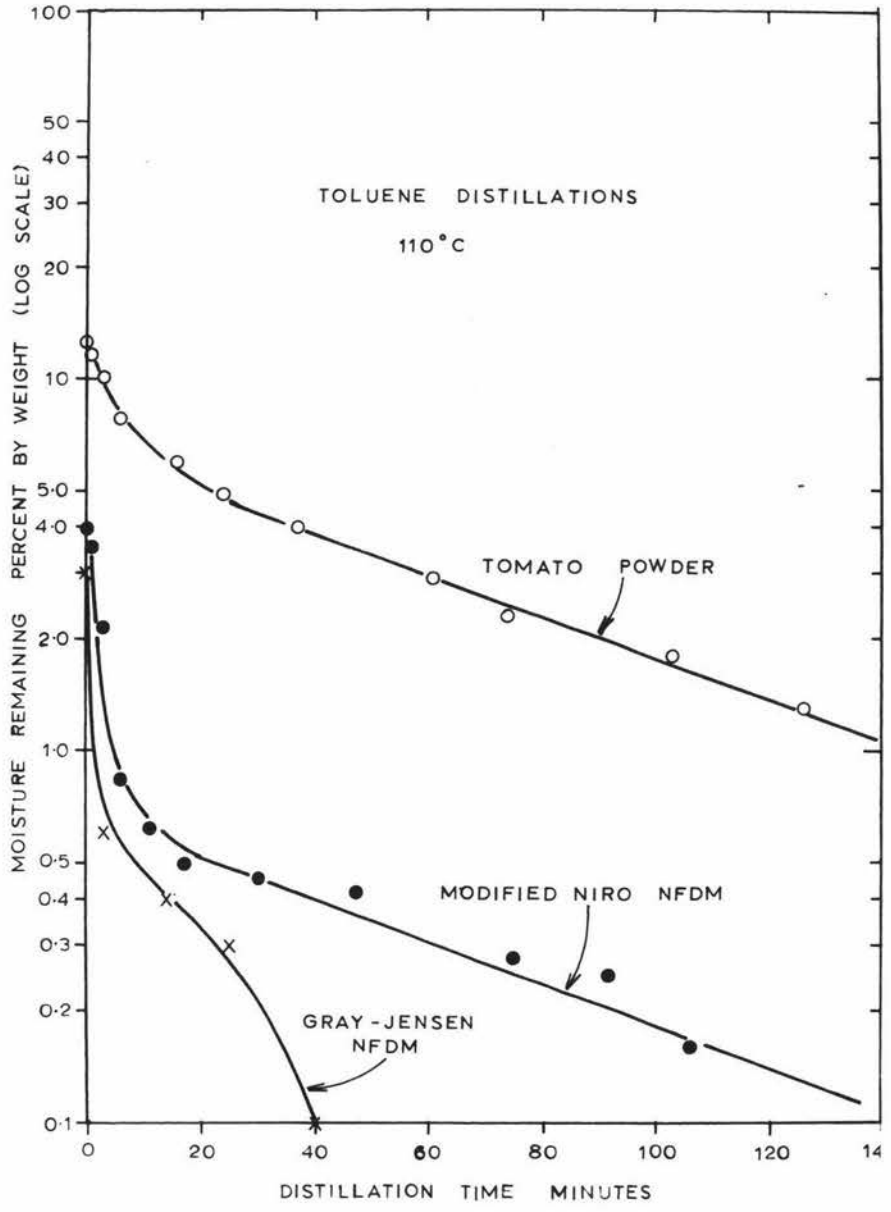


FIG. 5 REPRESENTATIVE DISTILLATION CURVES

tomato powder possibly contained other crystalline sugars. (Free sugars account for approximately 60% by weight of tomato solids, consisting mainly of D-fructose and D-glucose and a small amount of sucrose; Williams and Bevenue (1954)). All other samples were free of crystalline sugar, including two samples of instant NFDM.

In order to demonstrate the effect of the state of lactose in a milk powder upon azeotropic distillation rates, another approach was also used. A sample of Gray-Jensen NFDM was equilibrated at 55% R.H. for a period of several weeks so as to allow substantial lactose crystallisation. Equilibration was achieved over a saturated sodium dichromate solution. Toluene distillations were then performed upon the 55% R.H. NFDM and the control NFDM containing no crystalline lactose. The results are shown in Figure 6.

It is seen that with a toluene distillation the control NFDM containing no crystalline lactose showed complete moisture desorption within 40 minutes; whereas the NFDM with crystalline lactose required 70 minutes to reach the same stage.

However, this differential desorption rate was emphasised further by the use of a lower boiling point azeotropic boiling liquid. Benzene was used for this purpose, having a B.P. of 80°C, compared with toluene having a B.P. of 110°C. As can be seen from Figure 6 , using benzene distillation, the control NFDM required 60 minutes for moisture desorption whereas the 55% R.H. NFDM required approximately 130 minutes. Despite the longer distillation time required for benzene, the moisture content obtained for both samples corresponded with the toluene

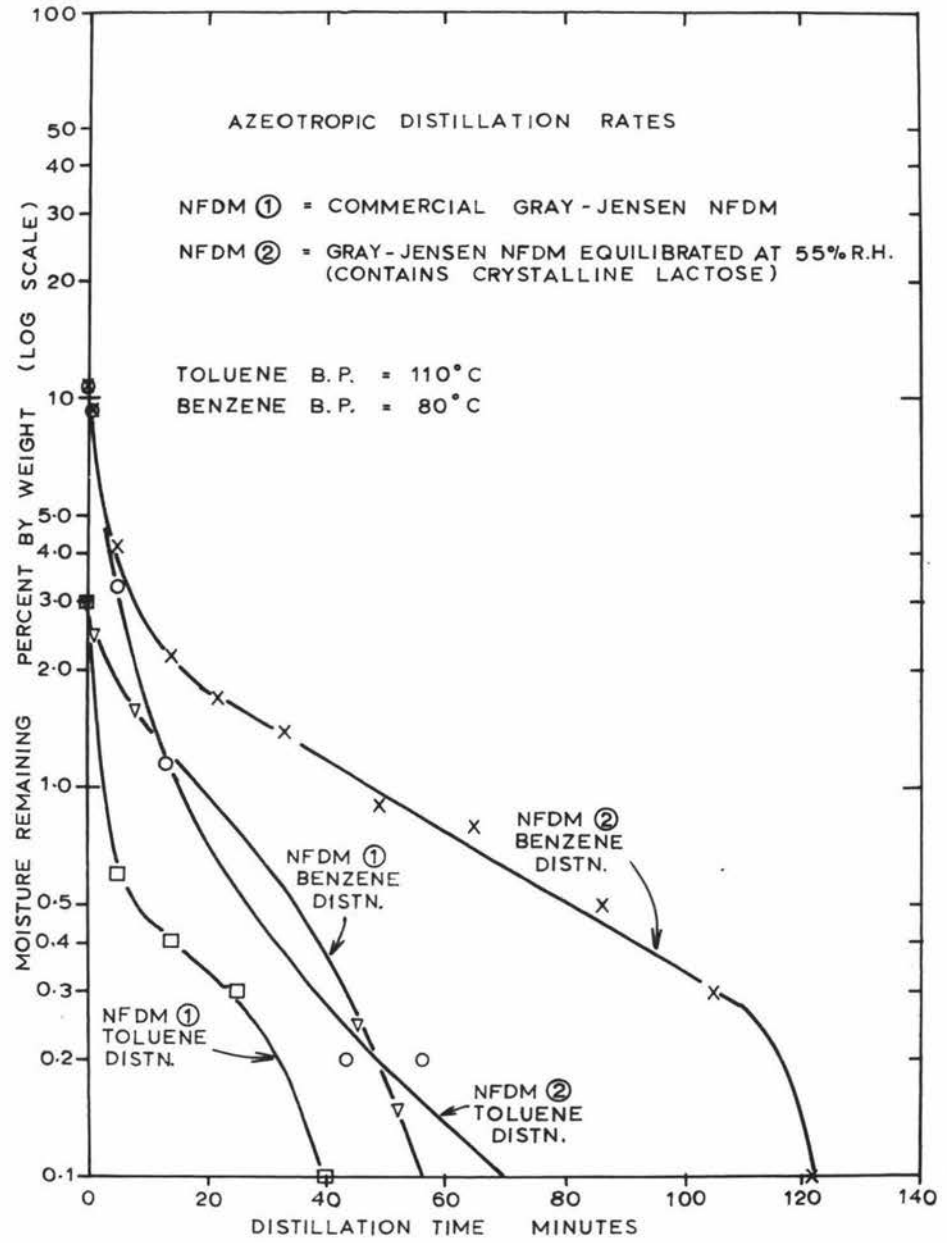


FIG. 6 EFFECT OF CRYSTALLINE LACTOSE UPON AZEOTROPIC DISTILLATION RATES

distillation result.

A theoretical explanation of these results is afforded by Choi et al (1948). They claim that the rate of dehydration of crystalline lactose hydrate follows a first order kinetic expression resulting in a straight line relationship when plotted on semi-log paper as in Figures 5 and 6. This bears out well with the dehydration rate curves obtained for samples containing crystalline lactose in this study. In every case where crystalline lactose was present in a sample a significantly longer distillation time was necessary in order to achieve complete desorption. It is reasonable to suggest that this technique may be useful as an index of the presence of crystalline lactose in NFDM. Since routine toluene distillation on NFDM are carried out in many laboratories then the presence of crystalline lactose would be suspected where an unusually long distillation time is required to achieve a constant reading.

In the case of the tomato powder interpretation of this observation is not as straightforward. Admittedly a semi-log plot of dehydration results in a definite straight line portion in the latter stages. This may be due to :

1. Desorption of water of hydration from sugars present at a rate determined by a first order kinetic expression; or
2. Caramelisation or browning of the sample to produce water. The curve obtained would suggest that such caramelisation follows a first order reaction. If this were the case, it

would be possible to extrapolate the straight line portion of the curve back to zero distillation time and find the quantity of free water originally present in the sample; as was shown by Choi et al (1948) for the case of lactose hydrate.

Further research in this field would appear worthwhile.

(ii) State of Lactose in Commercial NFDM

A quantity of Niro NFDM was obtained from a commercial factory for the purpose of research into the effect of rewetting processes upon the dispersibility of NFDM. In the course of this work it was desired to determine at which stage in remoistening lactose appeared in a crystalline state. Examination of samples revealed that, in fact, lactose occurred in a crystalline state in the original control powder obtained from the factory. This at first seemed rather surprising and it was originally thought that this may have been due to moisture pickup by the powder at some stage so as to allow crystal formation. This observation was, therefore, followed up by the examination of further samples from the factory; this time with a detailed summary of manufacturing conditions.

Samples of powder were obtained direct from the cyclone separator after drying and in this way could be matched up directly with a particular skim milk concentrate fed to the dryer previously.

Typical operating conditions at the factory were found to be concentration of the skim milk to approximately 48% total solids; the

temperature of the concentrate leaving the final effect of the evaporator being approximately 106°F. In addition, it was noted that the capacity of the evaporator is a little above that of the dryer, with the result that skim milk concentrate slowly accumulates over a period of several hours until the concentrate storage capacity of approximately 300gals is filled, when the evaporator is run on water for a time so as to allow the dryer to catch up.

It was found that powders produced under such typical operating conditions, consistently showed the occurrence of small lactose crystals. Powders produced from concentrate of lower total solids (e.g. 44%) and dried immediately without holding showed a much reduced incidence of crystalline lactose. Even at the high concentration to 48-49% total solids it was found that increased concentrate holding time had a marked effect upon increased incidence of lactose crystals, and upon their size.

A further observation was made which suggested that crystals were occurring in the powder as a result of self-nucleation of lactose in the skim milk concentrate. This was, that many small crystals were observed under the polarised light microscope to occur as separate identities, that is, not within a milk powder particle. In comparison, if a sample of milk powder which has been equilibrated at 55% R.H. for some weeks is examined, it will be found that considerable lactose crystallisation has occurred but in this case all crystals are contained within the skim milk powder particle.

An attempt was also made to examine the skim milk concentrate

directly for occurrence of crystalline lactose. It will be appreciated that this poses severe problems since any cooling of the concentrate or prolonged holding will favour self-nucleation and crystal growth and thereby introduce experimental artefacts. Unless a heated stage were employed on the microscope, some cooling could not be avoided. The way in which this was attempted, therefore, was to cool a weighed sample of concentrate of known total solids while simultaneously adding water to the concentrate so as to maintain the concentration of lactose in water at a point just slightly above the equilibrium solubility value for lactose at the corresponding temperature. This means that the concentrate is continually diluted while cooling so as to keep it only marginally supersaturated. When the concentrate reaches room temperature it can then be examined under the polarised light microscope for occurrence of crystalline lactose. Using this technique, lactose crystals were detected in concentrate of high total solids, but it is felt that more experimentation with this method would be desirable.

Theoretical considerations of the occurrence of crystalline lactose in a skim milk concentrate may best be explained by reference to Figure 7. (Lactose solubility figures from Whittier (1944)). Figure 7 relates the final or equilibrium solubility of lactose in water as a function of temperature. For example, at 110°F the final solubility is 36gm lactose per 100gm water. In addition a super-solubility curve is shown indicating the degree of supersaturation which may be achieved in a pure lactose/water system. Above this supersolubility value, self-nucleation would occur and crystal growth

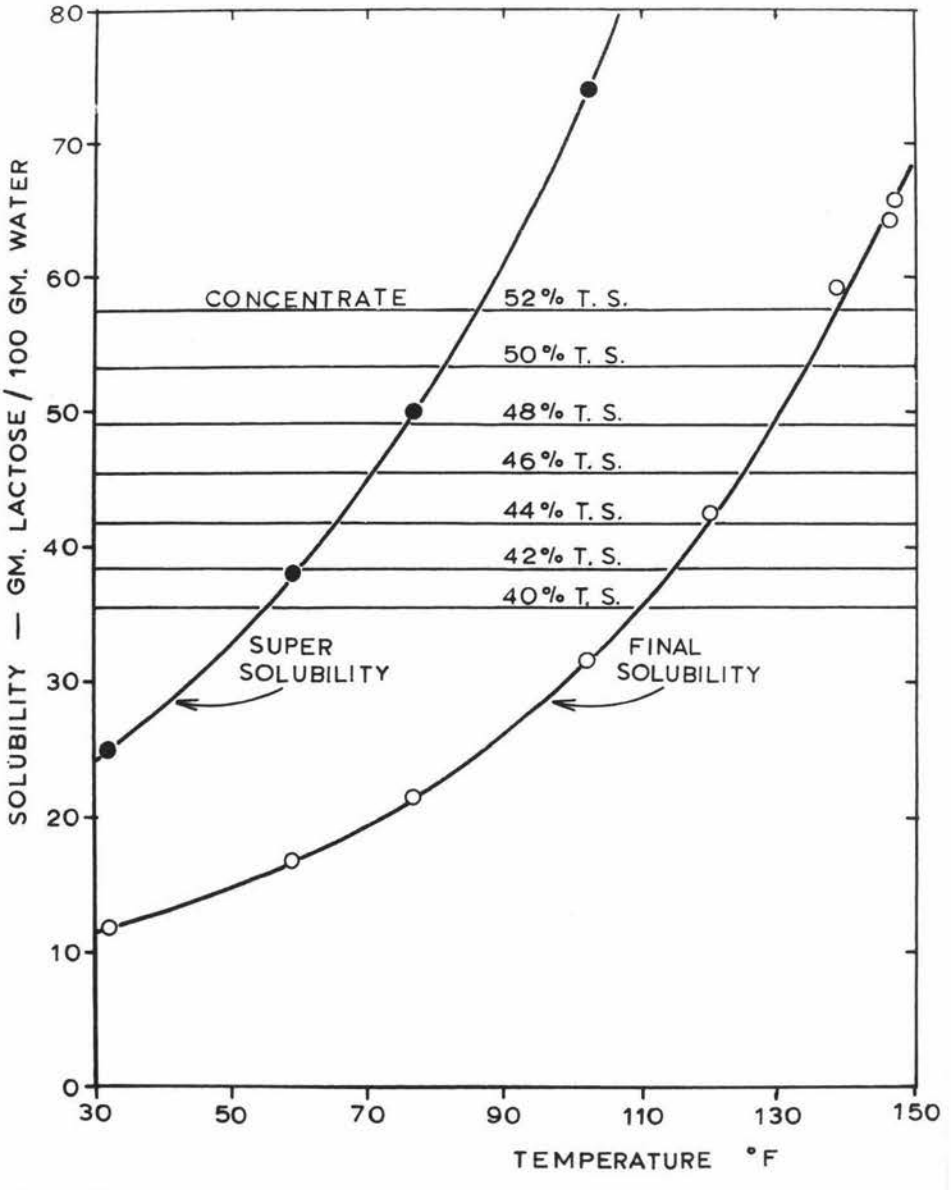


FIG. 7 SOLUBILITY OF LACTOSE AS A FUNCTION OF TEMPERATURE DATA OF WHITTIER (1944)

would continue until the final solubility value is reached corresponding to the temperature of the solution. Horizontal lines are also shown on Figure 7, representing the concentration of lactose in skim milk concentrates of varying total solids content, (assuming lactose to make up 53.2% of skim milk solids).

Therefore, taking a skim milk concentrate of, say 48% total solids, emerging from the last effect of the evaporator at 106°F, it will be seen from Figure 7 that the 48% concentrate will have a lactose concentration of 49gm per 100gm water. At this temperature the final solubility of lactose in water is seen to be 34gm per 100gm water. This means that the lactose in the concentrate is well into the supersaturated range. Hence, from a theoretical point of view it appears feasible that self-nucleation may be occurring, or that crystal growth is occurring around foreign nuclei such as clusters of casein micelles.

With recent advances in milk concentration, in particular the development of multiple effect falling film evaporators, concentration of skim milk to the range of 45-50% total solids is not uncommon. Also such concentrate is removed from the evaporator at relatively low temperatures, e.g. below 110°F. Both these factors, as can be seen from Figure 7, increase the probability of lactose crystallisation in the concentrate. It is believed that a stage has been reached where the occurrence of crystalline lactose in spray dried skim milk is no longer uncommon.

It is felt that the occurrence of crystalline lactose in

skim milk concentrate, without seeding offers several avenues for research. Among these may be listed :

1. Effect of crystalline lactose upon hygroscopic nature of NFDM. Sharp (1955) holds a patent employing the seeding of concentrate to produce this effect, claiming a less hygroscopic, more soluble and more dense powder. This principle is also employed in the drying of whey;
2. Effect of crystalline lactose upon the dispersibility of NFDM;
3. Effect of crystalline lactose upon particle characteristics; in view of the fact that the continuous lactose phase may be disrupted.

These observations of the occurrence of crystalline lactose in commercial NFDM have been reported fully in a paper by Morris, Neff, and Latimer (1967).

(iii) Working Theory of Mechanism of Dispersion

Eleven samples of food powders were analysed for dispersion and physical characteristics. These samples consisted of :

4 samples of spray dried NFDM

1 sample of spray dried Full Cream Dried Milk (FCDM)

2 samples of commercial instant NFDM

1 sample of roller dried NFDM

2 samples of spray dried instant coffee

1 sample of spray dried tomato powder

The results of these analyses are presented in Table 2. The type of drier employed for the milk powders is indicated in the results. "Modified Niro" represents a Niro spray drier modified to include fines recirculation into the atomising zone in an attempt to produce a "single stage instant".

Figure 8 shows microphotographs of several powders representing typical shapes and sizes encountered in such powders.

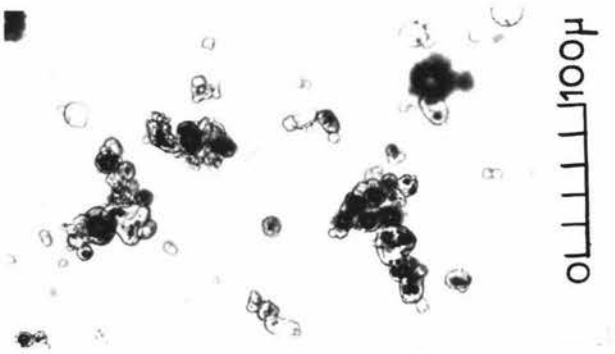
These results place in perspective the dispersibility characteristics and physical properties of such powders. Several interesting points are worth noting :

1. The samples of instant NFDM showed the highest dispersibility and sinkability properties and highest porosities;
2. The roller dried NFDM showed a moderate dispersibility and a high sinkability despite its low solubility (i.e. high solubility index). This can be attributed to the irregular shape of particles (as seen in Figure 8) and the high particle density;
3. The Coulter NFDM showed the best dispersion characteristics of all the spray dried NFDMs. This powder is produced by a special drier employing a venturi-atomiser for the dispersion of feed into the drying air stream; Townley and Coulter (1954); Bradford and Briggs (1963). As can be seen from Figure 8 this produces an agglomerated type product which may be termed a "single stage instant";

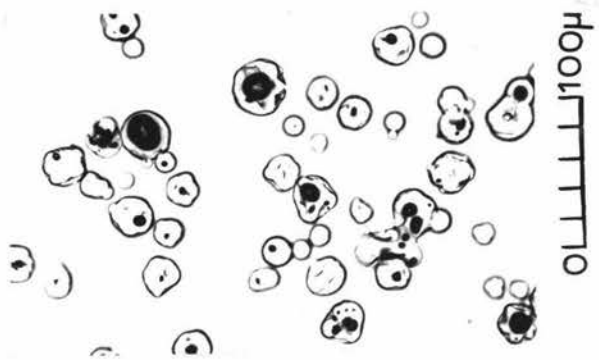
TABLE 2

Characterisation of Typical Food Powders

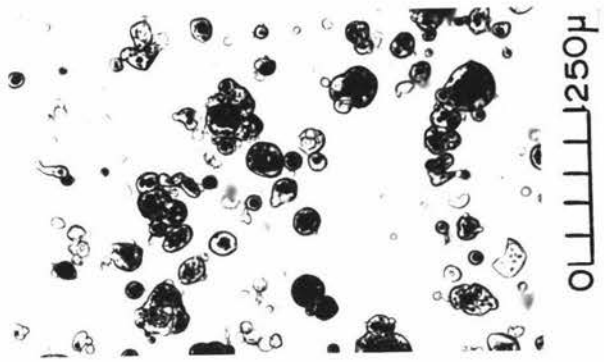
Sample	Moisture	Sinkability Index	Dispersibility	Solubility Index	HEXANE METHOD			Bulk Density (Tapped)	Geom. Mean Particle Size	Geom. Standard Deviation	Mean Aggregate Size	Lactose Crystallisation
					Bulk Density	Particle Density	Porosity (Loose)					
Modified Niro NFDM	3.98%	1.78mg/min.cm^2	28.5%	0.15ml	0.406gm/cc	0.85gm/cc	0.523cc/cc	0.498gm/cc	26 μ	1.96	-	+
Gray-Jensen NFDM	3.0	3.45	16.1	1.3	0.64	1.32	0.515	0.695	14	1.66	-	-
Rogers NFDM	3.6	5.2	16.4	0.1	0.58	1.25	0.535	0.62	<10	3.4	-	-
Coulter NFDM	4.3	5.5	39.1	2.2	0.59	1.21	0.510	0.715	22.5	2.05	-	-
Modified Niro FCDM	3.2	<0.20	11.2	4.5	0.448	0.88	0.490	0.435	21	-	-	-
Instant NFDM A (Base = Buflovak NFDM)	7.42	20.7	62.6	1.0	0.396	1.225	0.678	0.497	19	2.0	100 μ	-
Instant NFDM B (Base = Buflovak NFDM)	5.87	-	59.1	2.0	0.382	1.16	0.672	0.443	24	2.17	80	-
Roller Dried NFDM	3.45	12.0	31.5	13.5	0.503	1.46	0.655	0.64	22	3.3	-	-
Spray Dried Instant Coffee A	3.35	5.2	44.3	0.05	-	0.614	-	0.264	80	1.93	-	-
Spray Dried Instant Coffee B	2.32	4.6	28.7	<0.05	-	0.72	-	0.274	38	2.1	-	-
Spray Dried Tomato Powder	12.7	∞	-	-	0.67	1.43	0.535	0.68	54	1.66	180	-



Instant NFDM B
(Magnification x 250)

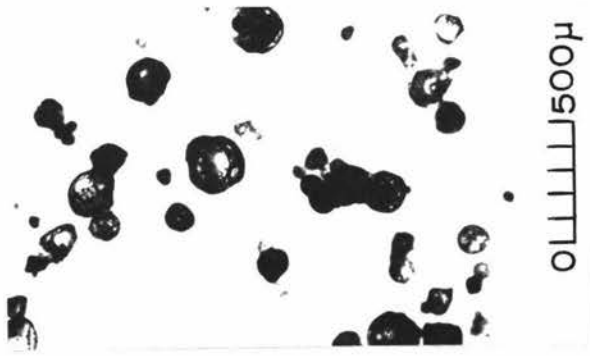


Gray-Jensen NFDM
(Magnification x 250)



Coulter NFDM
(Magnification x 100)

Roller Dried NFDM
(Magnification x 100)



Spray Dried Instant Coffee A
(Magnification x 40)

FIGURE 8 Microphotographs of Some Typical Powders

4. The Modified Niro FCDM showed an extremely low dispersibility and virtually zero sinkability. This can be attributed to the poor wettability resulting from the presence of fat. The effect of fat upon wettability and reconstitution is comprehensively dealt with by Pyne (1961);

5. The samples of instant coffee showed only moderate dispersibility and sinkability properties, in fact, in the same range as many spray dried NFDMs. Yet, instant coffee is generally looked on as a good example of an instant food powder. One reason for this is that in the reconstitution of a cup of instant coffee the average consumer would reconstitute to only a little above 1% solids whereas milk powder would be required to be reconstituted to approximately 12% solids. The dispersibility test mixes components at an equivalent of 11.5% solids;

6. In the case of the four samples of spray dried NFDM, the influence of particle density upon sinkability can be seen. The Modified Niro NFDM having a particle density of less than 1gm/cc showed a very low sinkability since dispersion would need to occur at the powder/water interface;

7. The instant coffee samples possess a very low particle density (0.6-0.7gm/cc) and yet showed moderate dispersibility and sinkability. Even though dispersion must occur at the powder/water interface, the moderate dispersibility can be attributed to the higher rate of solution of instant coffee

as compared to NFDM;

8. Subjective assessment of the tomato powder indicated excellent reconstitution properties. Due to the high viscosity of the reconstituted product the dispersibility test, as such, could not be employed.

As can be seen a broad range of food powders have been examined. While the results do not make possible direct correlations of factors responsible for dispersion upon reconstitution they, nevertheless, allow a better appreciation of the mechanism of dispersion. This, together with a literature review has made possible the formulation of a general working theory or hypothesis of the mechanism of dispersion. Pyne (1961) put forward a hypothesis of the importance of capillary movement of liquid in reconstitution, and then went on to supply experimental evidence in support of this hypothesis. His theory applied only to certain types of milk powder. It is intended here to give a more general hypothesis applicable to the reconstitution of any food powder.

It is postulated that the mechanisms of reconstitution can best be considered in terms of three major factors :

1. The Wettability-Capillarity of the Powder

This is obviously the first step since a particle must be wetted before it can be reconstituted.

For example, FCDM shows poor dispersion due, no doubt, to poor wetting of the particle surface. Tomato powder on the other hand

possesses infinite sinkability indicating a high wettability.

However, the shape or aggregation of particles will also have an influence on wettability-capillarity characteristics. This fact is illustrated in the case of instant NFDM where the dispersibility and sinkability of a powder is markedly increased by a process of agglomeration. This improvement in reconstitution is due, in part anyway, to a capillarity effect upon contact with water. Such improvement in wettability-capillarity is also noted in the case of roller dried NFDM.

Particle size appears to have a marked effect upon wettability-capillarity. Pyne (1961) showed that below a minimum particle size (9/u in the case of NFDM) no wetting of a powder surface occurred. Later work in this research also illustrates the very marked effect of granule or "particle" size upon both sinkability and dispersibility. To some extent this can be explained by the effect of particle size upon the surface area to mass ratio (or upon hydraulic radius). As particle size is increased, the surface area per unit mass to be wetted is decreased thus tending to improve the wettability characteristics.

2. The Particle Density of the Powder

Particle density may influence reconstitution in two ways. Firstly, a high particle density will tend to increase the wettability of a particle. In other words, the ability of a particle to be wetted when placed at the surface of a liquid will be a function of its surface area, its inherent wettability (chemical composition) and

its density.

Secondly, particle density will influence reconstitution by determining the ability of a particle to separate or sink from the surface when wetted. If a wetted particle possesses a density greater than that of water, 1gm/cc, then it will separate from the unwetted powder mass. This must be considered desirable since dispersion is more easily effected when particles are distributed throughout the volume of water. This fact was demonstrated by Harper et al (1963) when they studied the effect of concentration of milk solids in the vicinity of the particle upon its instant solubility.

Where a powder possesses a particle density less than that of the reconstituting liquid then dispersion must occur at the powder/liquid interface. This tends to promote caking of the powder mass, reconstitution then being dependent on the rate of solution at the interface. This particular situation is well illustrated by instant coffee.

3. Rate of Solution of Substance

The last phase in reconstitution will be the rehydration or "solution" of the particles. The rate at which this final state is achieved will influence the dispersibility of the powder.

A high rate of solution can impart reasonable dispersibility to a powder having poor wettability or limiting particle density. This is illustrated by the case of instant coffee, where all dispersion must occur at the water/powder interface but due to a high rate of

solution, a reasonable dispersibility is achieved.

It is interesting to note that rate of solution will be directly proportional to the surface area to mass ratio, whereas wettability is inversely proportional.

Rationalising the mechanism of dispersion in terms of these three major factors, wettability-capillarity, particle density and rate of solution, it is possible to construct certain models in order to represent any food powder.

Examples of such models are represented diagramatically in Figure 9.

Model 1

Low Wettability-Capillarity, Low Particle Density, Medium Rate of Solution

Here the first stage in dispersion, viz. wetting of the particles, will be a limiting factor. Even when a particle is wetted its low density will prevent separation from the interface. Dispersibility and sinkability of such a substance will, therefore, be poor; being dependent on the rate of solution from the interface.

Certain milk powders would appear to fall into this category in particular spray dried FCDM and some NFDMs.

Model 2

Low Wettability-Capillarity, High Particle Density, Medium Rate of Solution

This illustrates the effect of a higher particle density in allowing separation from the interface once a particle is wetted. Dispersibility of such a system will be greater than Model 1. Low

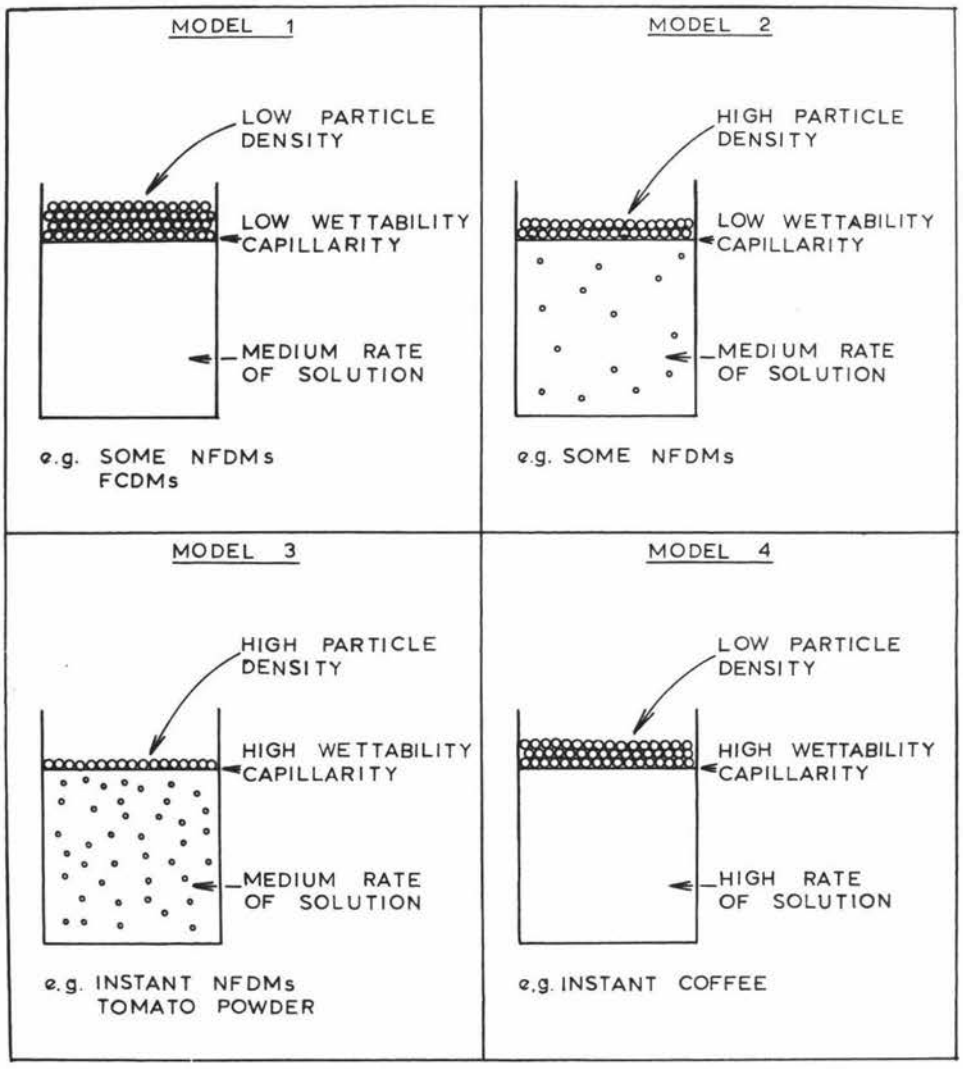


FIG. 9 MODEL REPRESENTATION OF MECHANISM OF DISPERSION OF A POWDER

wettability-capillarity is still a limiting factor.

Certain spray dried NFDMs would fall into this category.

Model 3

High Wettability-Capillarity, High Particle Density, Medium Rate of Solution

Such a system would show marked improvement in dispersibility over Models 1 and 2. The only limiting factor would be the rate of solution of the substance.

Into this category could be classed instant NFDMs and tomato powder.

Model 4

High Wettability-Capillarity, Low Particle Density, High Rate of Solution

This example shows the limiting effect of a low particle density. The particles cannot separate into the liquid and dispersion must take place at the interface. Dispersibility is then dependent on the rate of solution.

Spray dried instant coffee would fall into this category.

Obviously, other combinations are possible, and further models may be constructed. However, these four models serve to illustrate the manner in which a powder can be categorised so as to elucidate the mechanism of dispersion.

It is believed that such illustration considerably simplifies an understanding of the mechanism of dispersion of a powder. In this way it is now possible to extend such principles in an effort to improve the dispersibility of typical powders.

D. PRINCIPAL RESEARCH FINDINGS

1. It has been demonstrated that the presence of crystalline lactose in a milk powder will influence the rate of moisture desorption upon azeotropic distillation. For example, a toluene distillation time in excess of 60 minutes is required for complete moisture removal from a sample containing lactose in a crystalline state; such observation may well be employed as an index of state of lactose during routine analysis;
2. It has been found that lactose may exist in a crystalline state in commercial spray dried NFDM, such observation having been correlated with manufacturing conditions;
3. Theoretical considerations of lactose solubility data as compared with lactose concentrations in skim milk concentrates reveal the strong probability of lactose crystal nucleation and growth occurring in such concentrate, particularly in view of the high solids concentrations and low holding temperatures employed today;
4. A working theory has been proposed to explain the mechanism of dispersion of a food powder. This theory takes into account the wettability-capillarity characteristics, the particle density, and the rate of solution of the powder;
5. It is shown how by means of this working theory it is possible to construct models to represent the mechanism of dispersion of any food powders.

III. EXPERIMENTAL WORK

SECTION (2) EFFECT OF NON-REWETTING PROCESSES UPON DISPERSIBILITY

SECTION (2) - EFFECT OF NON-REWETTING PROCESSES UPON DISPERSIBILITY

A. INTRODUCTION

It is appreciated that considerable improvements in the dispersibility of a powder are to be achieved by such techniques as instantising of NFDM. However, instantising type processes involve the rewetting of a base powder to achieve agglomeration of particles, followed by redrying; such operations adding considerably to the complexity and cost of manufacture. It was, therefore, decided to investigate the possibilities of improving dispersion characteristics of a food powder by non-rewetting processes.

In addition, it was desired to relate and explain any changes observed in reconstitution characteristics of powders in terms of the working theory of dispersion already developed.

Keeping in mind that this was to be an attempt to improve reconstitution by non-rewetting processes it was decided to study the effect of compression of powders. Such a study appeared justified on referring to the working theory of dispersion and noting the following points :

1. The importance of capillary characteristics of a powder. It appeared feasible that compression could promote aggregation of particles which would result in more rapid capillary penetration of liquid upon reconstitution;
2. The importance of particle density. Remembering that many spray dried particles contain entrapped gases it would seem reasonable that the particle density of a powder could be

increased by compression. The effect of this upon dispersibility was worthy of investigation.

The description and presentation of research dealing with non-rewetting processes is best classified into three headings, viz. :

(i) Compression of powders;

(ii) Temperature of water for reconstitution;

(iii) Addition of free flow agents.

B. LITERATURE REVIEW

(i) Compression of Powders

Many workers have reported on the effect of compression of milk powders upon bulk density and residual oxygen content, but seldom is there any mention of the effect upon reconstitution characteristics. Webb and Hufnagel (1943) reported on the saving in shipping space which could be achieved by compression. They related pressures, volume reduction and packing densities for NFDM and FCDM. Similar work is reported by Miller (1945). Lea et al (1943) and Thiel (1945) extended this work by studying the effect of compression upon the residual oxygen content of FCDM. It was hoped that the milk powder could be compressed sufficiently to expel oxygen responsible for oxidative deterioration thus eliminating the need for inert gas or vacuum packing of FCDM. Although residual oxygen contents could be reduced to levels comparable to those achieved by gas packing this technique has never been accepted commercially.

Brochner (1962) and Arbatskaya (1962) report on the

manufacture of dried milk tablets employing compression of constituents.

Wagner et al (1964) report a compression technique employed for foam mat dried orange and grapefruit juice powders involving heating/compression of the powder in a modified double drum drier. This technique produced a more acceptable powder of increased bulk density although it is reported that reconstitution time suffered slightly.

Hanrahan and Kontson (1965) studied the effect of compressing NFDM into cake form upon bulk density of the cake and upon bulk density and dispersibility of the repowdered sample. These studies were made on commercial spray dried NFDM, instant NFDM, and foam spray dried NFDM. It was found that compression adversely affected the dispersibility of the repowdered samples in all cases except at very low pressures (up to 600 psi) in the case of foam spray dried NFDM. This work is the only report in the literature of the effect of compression upon dispersibility. It appeared warranted to extend this work in order to study the effect of compression upon particle density and porosity of the powder; also to apply the technique to two different classes of spray dried food powders.

Further support, suggesting the use of compression as a means of achieving agglomeration comes from a study of practice in the pharmaceutical industry and powder compaction in other industries; Peck (1958); Train and Lewis (1962); Gregory (1962); Little and Mitchell (1949). Granulation is a size enlargement operation employed for example in the pharmaceutical industry, in the fertiliser industry, and in metallurgy. One method of granulation sometimes employed in

the pharmaceutical industry is termed "dry granulation" or"slugging". This involves the "slugging" or compression of the base powder to form oversize tablets, followed by size reduction to the desired granule size. The overall result is one of size enlargement or agglomeration by compaction.

(ii) Temperature of Water for Reconstitution

It is appreciated that the temperature of the water of reconstitution will influence the rate of dispersion of food powders. However, optimum temperatures of reconstitution have seldom been determined. Instructions for reconstitution found accompanying food powders will often recommend the use of cold water with little justification.

Work is reported on the effect of water temperature in the reconstitution of milk powders. King (1966) reviews much of this work. Ashworth and Bendixen (1947) report that there exists an optimum temperature for reconstitution of milk powders but fail to give an actual value. Aldrich and Downs (1959) used a manual stirring dispersibility test to determine the effect of reconstitution temperatures in the range 50°C to 100°C upon dispersibility. They tested instant NFDMs, spray dried NFDMs and FCDMs, and found in all cases that increasing water temperature above 50°C decreased dispersibility. Gibson and Raithby (1954) found that optimum temperature for wettability (equivalent to sinkability in present work) was 50°C for NFDM. Above 50°C there occurred a sharp decrease in the wettability of the powder.

The decrease in dispersion or wettability at higher temperatures is generally attributed to a denaturation of casein. This corresponds well with work of Howat and Wright (1933). They determined the percentage of insoluble protein in milk powders (NFDM and FCDM) after reconstitution at different temperatures. For roller dried samples it was found that a minimum percentage of insoluble protein was obtained at approximately 45°C. This effect was not obtained for spray dried powders.

Although work is reported on the effect of temperature of reconstitution upon rate of reconstitution of milk powders there appears to be some confusion as to optimum temperatures. This fact is evidenced in a review by Gibson (1952) describing industrial methods for reconstitution. Methods show great variation in water temperature employed ranging from 45-120°F. It appeared desirable, therefore, to study the effect of water temperatures, at least for NFDM.

(iii) Addition of Free Flow Agents

Interest in free flow agents with regard to this study can be said to be twofold :

1. It is noted that instant milk powders possess improved flow properties as a result of increased particle size. The question arises, therefore, whether some of the improvement in dispersibility of instant powders is due to improved flow properties?

2. It is noted that the flow properties of food powders can be improved by chemical agents. It is desired to determine the effect of such agents upon the dispersibility of the powders.

Good reviews of flow properties of powders and methods of measurement are given by Carr (1965) and Burak (1966).

Burak (1966) states that flow difficulties become acute at particle sizes below about 100/u. This, of course, includes spray dried NFDM and many other food powders. However, flow characteristics can be greatly improved by the addition of certain chemicals loosely termed free flow agents and anticaking agents. The mode of action of such free flow agents can be put into two main groups :

1. Chemical agent which physically separates powder particles to prevent bridging. Such an agent may act as a lubricant between particles to improve powder flow;
2. Chemical agent which selectively absorbs moisture in the environment to prevent caking. Such an agent is truly an anticaking agent.

Many free flow agents combine both these modes of action.

Watson (1957) describes the use of anticaking agents in the conditioning of table salt. Sjollema (1963) reports on some investigations of the free flow properties of milk powders and use of free flow agents. Linton-Smith (1961) and Linton-Smith and Hansen (1963) report on the use of free flow agents in improving the flow

properties of milk powder for use in hot drink dispensing machines. These agents also serve in reducing the packing volume of powders.

Free flow agents which have been used or proposed for powders include synthetic silicas, silicates, and metallic oxides, carbonates and phosphates. Flow-conditioning agents or lubricants are also employed in the pharmaceutical industry to assist tabletting; common agents being talc and magnesium stearate. In general, the flow-conditioning agent should have a particle size very much below that of the host powder.

It should be remembered that improved flow properties in a powder will, of itself, be a desirable quality attribute quite apart from any effect an additive may have on other powder properties, such as dispersibility.

C. PROCEDURE FOR COMPRESSION

Compression of powders was achieved by means of a hydraulic press and a simple punch and die set. The hydraulic press was operated by compressed air, fed into a pressure chamber having a bore diameter of 12 inches, (113 inches^2 area). This means that for every 1 psi of air pressure in the pressure chamber, there will be a downward force of 113lb.

It was realised in the course of experimentation that the downward force, as calculated from the air pressure admitted to the chamber, did not correspond with the downward force shown by the punch. This is due to an upward force exerted by an internal spring acting to

retract the piston of the press when air pressure is released, i.e.

$$\begin{pmatrix}\text{Downward Force}\\ \text{of Punch (lb)}\end{pmatrix} = \begin{pmatrix}\text{Pressure in}\\ \text{Chamber psi}\end{pmatrix} \times 113 - \begin{pmatrix}\text{Upward Force of}\\ \text{Spring (lb)}\end{pmatrix}$$

Since the force exerted by a spring is a function of its compression or elongation, this factor will be variable depending upon the distance of travel of the piston, i.e. Hooke's Law.

Therefore, this necessitated the calibration of the retracting spring, as a function of distance of travel of the press piston. This was done by :

1. Calculating total downward force from air pressure in chamber;
2. Measuring the net downward force on punch by means of a spring balance attached to a beam above the hydraulic press;
3. Measuring the distance of projection of the piston;
4. Calculating the upward force of the spring as per formula shown.

This then allowed plotting of a calibration curve shown in Figure 10. As can be seen, Hooke's Law is demonstrated from the straight line plot obtained. Using Figure 10 it was possible to determine the net downward force on the compression punch from the air pressure admitted to the chamber and the distance of projection of the piston. The net force on the punch is converted to psi from a knowledge of the punch face area. Two punch and die sets were employed having punch face areas of 2.0 inches^2 and 3.73 inches^2.

The procedure adopted for the compression of NFDM powder and instant coffee powder was as follows :

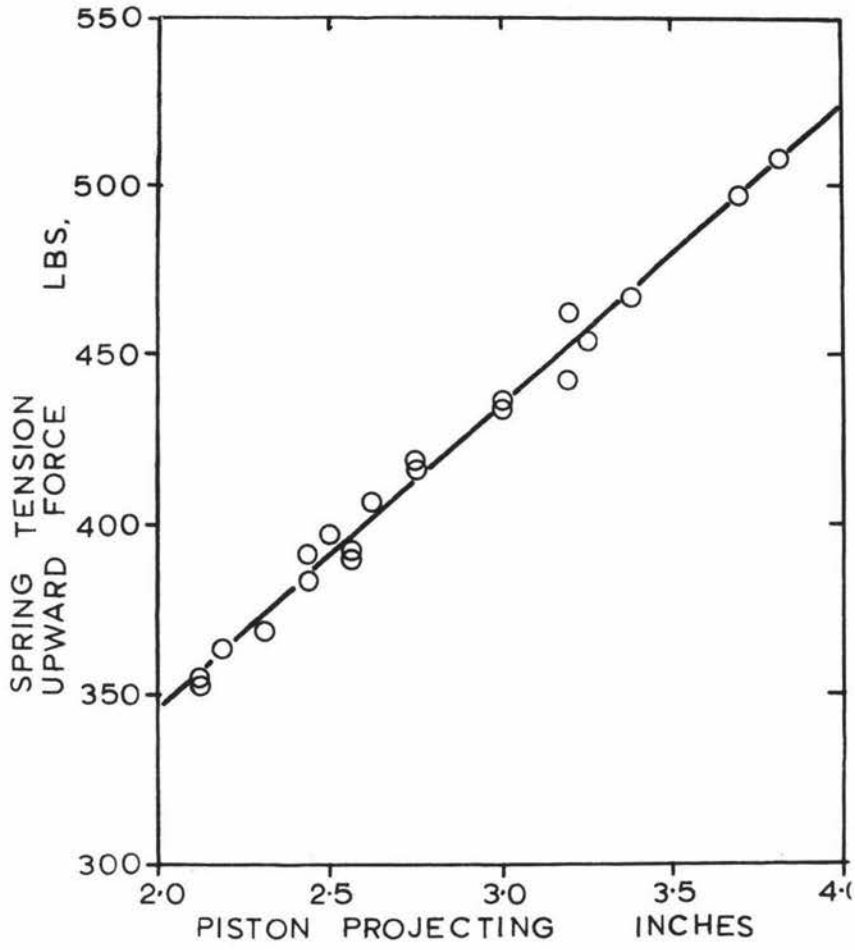


FIG. 10 CALIBRATION OF HYDRAULIC PRESS

1. The 2.0 inches^2 area punch and die set was used for NFDM samples while the 3.73 inches^2 area set was used for instant coffee samples. In both cases loose powder depth of fill in the dies was approximately $\frac{7}{8}$ inches. Samples were compressed at varying pressures, pressures being calculated as described above. Compressed sample was immediately transferred to a closed jar, each operation being repeated until sufficient sample for test was obtained;
2. The samples at each compression pressure were transferred to a "Moulin" manual kitchen grinder. This served to break up samples by forcing particles through a perforated plate. In all work the smallest plate having 1,400/u holes was employed;
3. For the sake of uniformity, samples were next screened through a single standard sieve. For NFDM, a 36 mesh B.S. sieve (420/u) was employed while for the instant coffee an 18 mesh B.S. sieve (850/u) was used;
4. Since all the powders employed are extremely hygroscopic, care was taken throughout to minimise moisture uptake. Moisture checks revealed that the complete compression and regrinding operations resulted in a moisture uptake from 3.6% to 5.0% for NFDM and from 2.8% to 3.6% for instant coffee. This minimal moisture uptake was considered acceptable.

D. RESULTS AND DISCUSSION

(i) Compression of Powders

(a) Non-Fat Dried Milk (NFDM)

Samples of Niro NFDM were prepared by a process of compression and regrinding as described under PROCEDURE. Pressures of treatment varied from 0-6,000 psi. In addition to commercial Niro NFDM, the study was also repeated with Niro NFDM + 2% Magnesium Stearate powder added in the dry state. Magnesium Stearate is commonly employed as a lubricant and free flow agent and as an aid in compression in the pharmaceutical industry. Treated samples were then analysed for dispersibility, sinkability, particle density, bulk density and porosity. Microscopical examination was also carried out.

The results are best presented in graphical form and are shown in Figure 11. The following points are noted from the results :

1. No significant improvement in dispersibility can be achieved in NFDM by compression. Beyond 1,500 psi the dispersibility of the reground powder shows a steady decrease; whereas up to 1,500 psi there is little effect upon dispersibility;
2. The sinkability of the Niro NFDM shows a slight increase on compression but not until the particle density of the powder had exceeded approximately 1.18gm/cc;
3. It is clearly demonstrated that the particle density of spray dried NFDM can be increased by compression. This is due to the unique nature of spray dried powders. Since particles contain entrapped gas cells it is possible to decrease

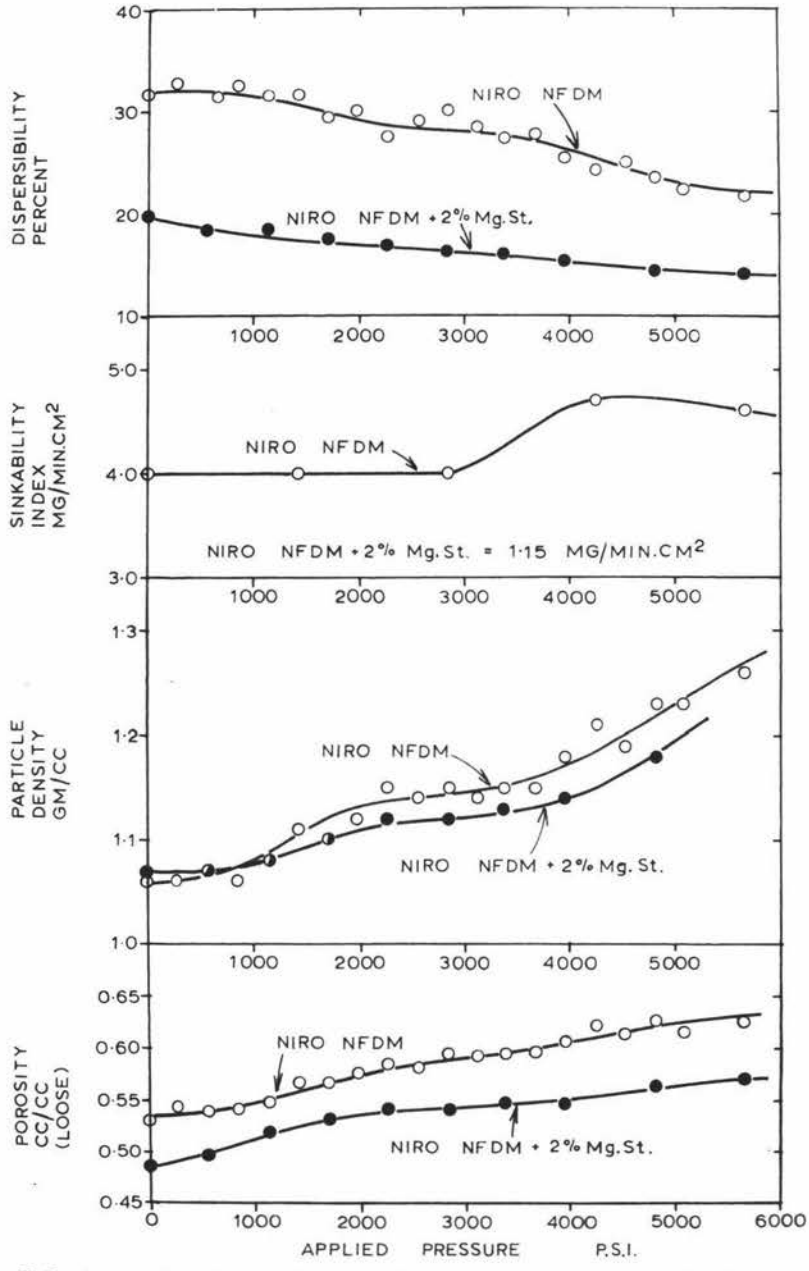


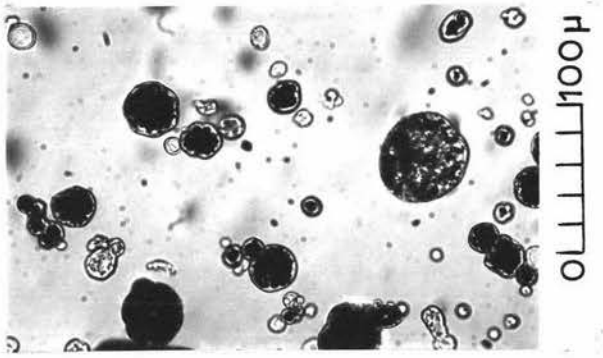
FIG. 11 COMPRESSION OF NIRO NF DM (REPOWDERED)

particle volume by compression;

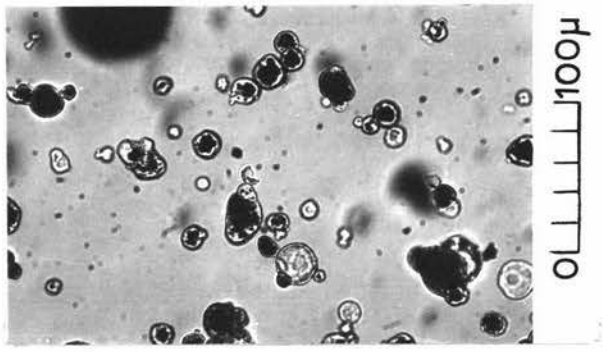
4. An interesting effect of compression is the increase in porosity which can be achieved. Porosity is expressed as cc of voids per cc of loose powder. This increase in porosity could be achieved either by a tendency of particles to aggregate or by fragmentation to form irregular particles. Microscopic examination revealed that, in fact, both effects were occurring. At low pressures considerable tendency for particle aggregation was evidenced. However, the effect of higher pressures was merely to increase fragmentation of particles. This effect of compression is well illustrated by the microphotographs in Figure 12;

5. The bulk densities (loose) of the regound powders showed little change and have not been plotted. Bulk densities of the Niro NFDM ranged only from 0.50-0.47gm/cc for the pressure range 0-6,000 psi. Correspondingly, the bulk densities with 2% Magnesium Stearate added ranged from 0.55 to 0.50gm/cc. It should be noted that bulk densities can be calculated from the particle density and porosity figures which have been plotted;

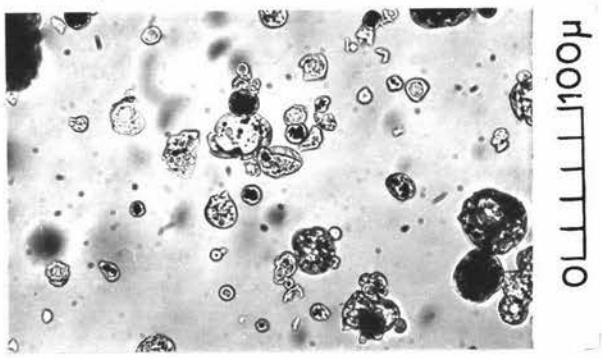
6. The effect of Magnesium Stearate addition upon compression of NFDM provides an interesting comparison. Firstly, it must be mentioned that, subjectively, NFDM with 2% Magnesium Stearate appeared far superior in free flow properties and general handling characteristics. The compression, re-grinding, and sieving operations were all facilitated with this additive.



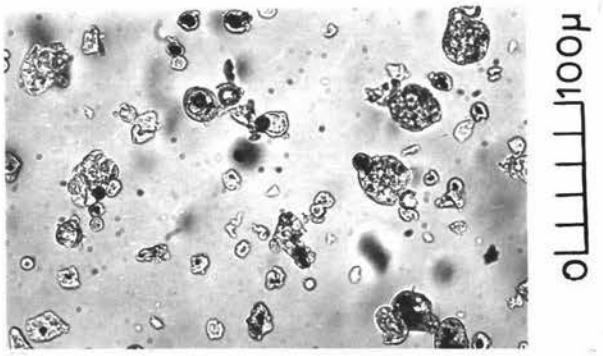
Control : 0 psi



Compression : 570 psi



Compression : 2550 psi



Compression : 5090 psi

FIGURE 12 Microphotographs Showing Effect of Compression/Repowdering Treatment upon Niro NFDM (Magnification x 250)

However, the effect upon dispersibility was far from beneficial. This can be explained by the hydrophobic properties of the Stearate which in effect has given the NFDM particles a coating as for FCDM. This is reflected in the low sinkability value with Stearate addition, reducing sinkability index from 4.0 to 1.15mg/min cm^2 at 0 psi;

7. The lubricating effect of Stearate during compression is evidenced in the plot of particle density versus applied pressure. As can be seen in Figure 11, at any particular applied pressure the sample with Stearate added will have undergone a smaller increase in particle density than the straight Niro NFDM. This is attributed to the lubricating effect of the Stearate in allowing better dissipation of compression energy;

8. The role of Stearate as a flow-conditioner is seen in the porosity measurements. Reground samples with this additive consistently show a lower porosity indicating greater packing of the powder;

9. The highest particle density achieved in this study is 1.26 gm/cc at 5,650 psi. At 0 psi the control NFDM possesses a particle density of 1.06gm/cc. Buma (1965) discusses and reviews work on the determination of the true density of dairy products. He arrives at a value of 1.52gm/cc for the true density of NFDM solids. Accepting this figure it is seen that compression has effected 43.5% of the maximum

theoretically possible increase in particle density.

From these results it is concluded that no significant improvement in the dispersibility of spray dried NFDM can be achieved by compression. In itself, this conclusion is no different to that obtained by Hanrahan and Kontson (1965) except that in the present study no adverse effect on dispersibility was noted below 1,500 psi, whereas Hanrahan and Kontson found an adverse effect at lower pressures. This observation gives added feasibility to the suggestion that powders may be compressed for packaging and distribution in order to reduce packaging, storage and transport costs. As mentioned earlier, this has been suggested by many workers in the past but never with any reference of the effect of compression upon reconstitution characteristics.

However, these results and conclusions extend beyond those of Hanrahan and Kontson (1965) in so far as they have included measurements of particle density and porosity. When the results are examined with regard to the working theory of dispersion previously developed some interesting observations are possible.

Firstly, the influence of particle density upon sinkability of the powder has been clearly demonstrated. It is seen that an increase in sinkability is achieved beyond a particle density of 1.18gm/cc.

Secondly, it has been shown that the porosity of the powder can be increased by compression. This porosity increase may be effected by :

1. Aggregation of particles as a result of particle/particle adhesion;

and/or

2. Fragmentation of particles to form irregular shaped units.

Microscopic examination suggests that both effects are occurring although fragmentation appears predominant at higher pressures, (e.g. Figure 12). It would be expected that since the porosity of the powder is increased the dispersibility should be improved due to increased wettability-capillarity, and yet this was not the case. This can be attributed to the decrease in particle size associated with fragmentation. Pyne (1961) illustrated the importance of particle size upon the wettability of particles. Later work in this thesis also emphasises the importance of particle size upon reconstitution characteristics.

(b) Instant Spray Dried Coffee (Brand A)

The study of effect of compression upon dispersibility of a food powder was extended to the case of Spray Dried Instant Coffee A. The main interest in this study is due to the low particle density of instant coffee, e.g. 0.65gm/cc. Characteristically, instant spray dried coffee particles contain a large volume of entrapped air, being virtually a hollow shell. Dispersion of such a powder must occur by solution at the powder/water interface, (Model 4). It appeared feasible, therefore, that compression could be employed to increase particle density so as to allow sinking of wetted particles from the surface and so facilitate reconstitution (Model 3).

Samples of Instant Coffee A were prepared by compression and regrinding as described under PROCEDURE. Pressures of treatment varied from 0-1,700 psi. Samples were analysed for dispersibility, sinkability, particle density, bulk density and porosity. Microscopical examination was also carried out.

Results are presented in graphical form in Figure 13. Before discussing these results it may be said that the immediate result which caught the eye was the slight, but significant, increase in dispersibility possible at relatively low compression pressures. This led to the repeating of the experimental procedure and analyses, but this time with greater emphasis on the range of compression from 0-350 psi. The results at this lower range of pressures are presented in Figure 14. The results are presented separately since a different batch of instant coffee A was used in this repeat trial.

The following points are noted from the results in Figures 13 and 14 :

1. A slight improvement in the dispersibility of this brand of spray dried coffee is achieved by compression below approximately 250 psi. Certainly, there is no adverse effect on dispersibility by such treatment, below 250 psi;
2. The effect of compression on sinkability is quite an interesting one. Initially, there occurs a decrease in the sinkability index until, at above approximately 350 psi, a gradual increase in sinkability is achieved. It is interesting to note that the sinkability does not show an

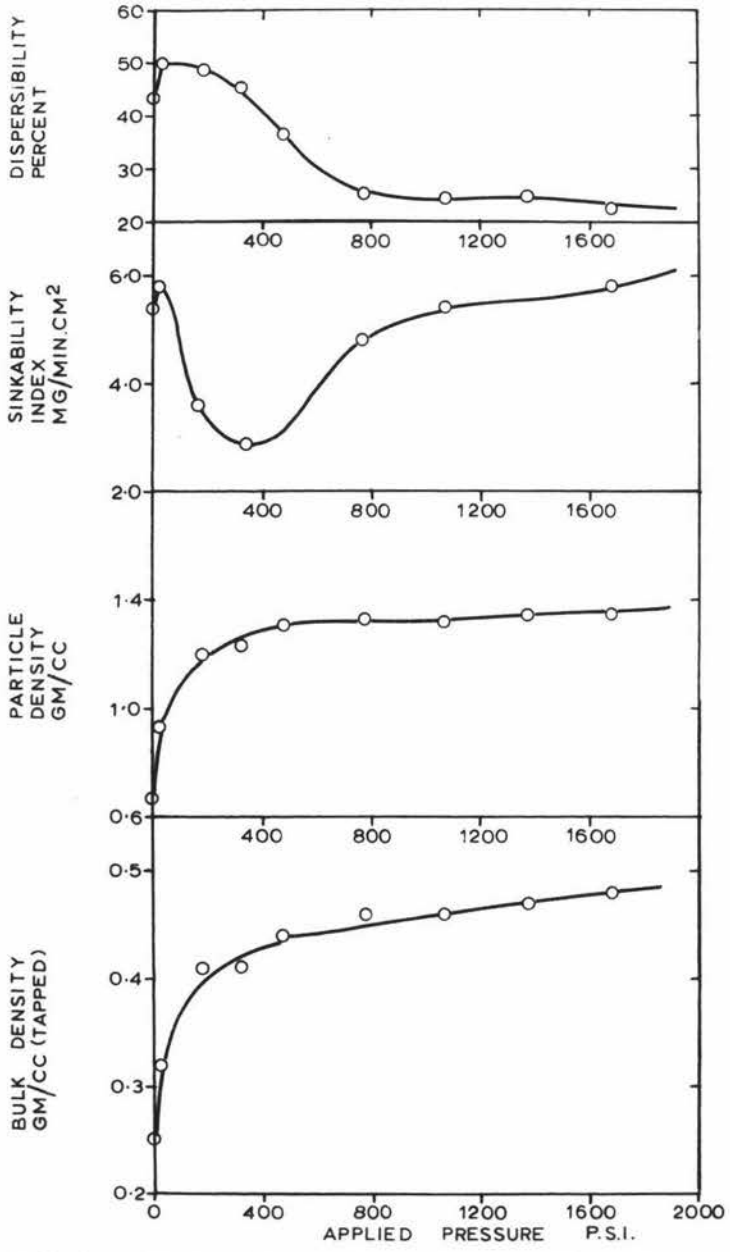


FIG.13 COMPRESSION OF INSTANT COFFEE A
RANGE 0 - 2000 P.S.I. (REPOWDERED)

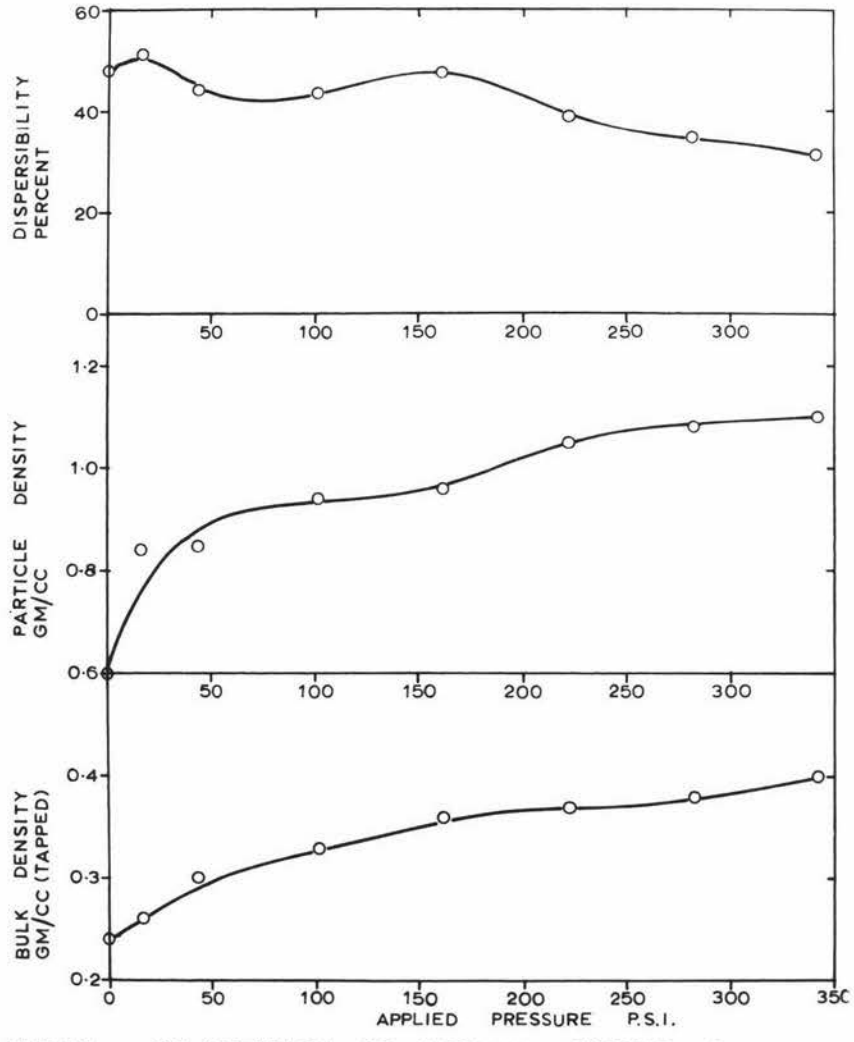


FIG.14 COMPRESSION OF INSTANT COFFEE A RANGE 0 - 350 P.S.I. (REPOWDERED)

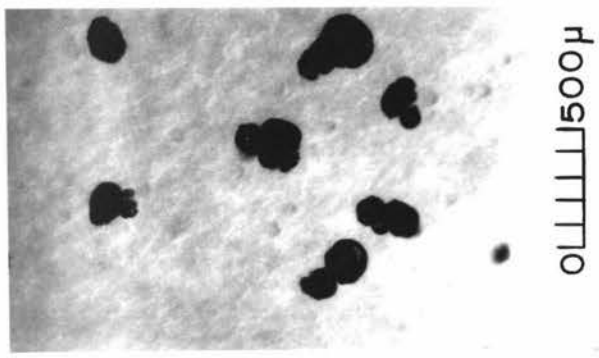
increase until the average particle density of the powder has reached 1.2gm/cc; this was also the case with NFDM;

3. With spray dried coffee the effect of compression upon particle density is much more marked than with NFDM. In fact, even at 600 psi, the particle density has almost doubled from 0.67 to 1.32gm/cc;

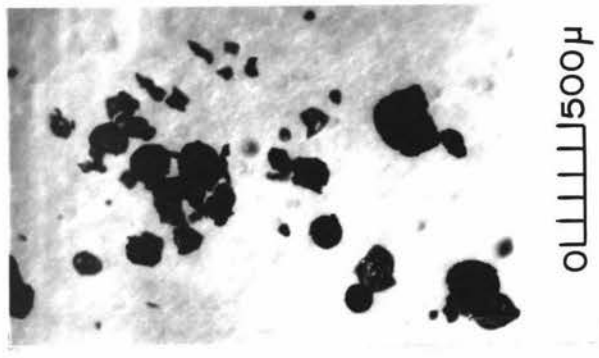
4. Microscopic examination revealed quite plainly that the effect of compression was to cause successive fragmentation of the originally hollow spray dried spheres. This can be seen in the microphotographs shown in Figure 15;

5. Another interesting effect of compression relates to the bulk density of the <u>reground</u> powder. It is well appreciated that commercial instant coffee possesses an extremely low bulk density, e.g. 0.24gm/cc in this instance. Any increase in the bulk density of such a powder would represent savings in packaging, storage and transport costs. Now, it is seen from Figure 14 that at, say, 150 psi compression treatment there is no adverse effect on the dispersibility of the powder, and yet bulk density of the reground powder has increased from 0.24 to 0.35gm/cc. This represents a potential saving in the packaging volume of the present product of 31%. Such a saving speaks for itself;

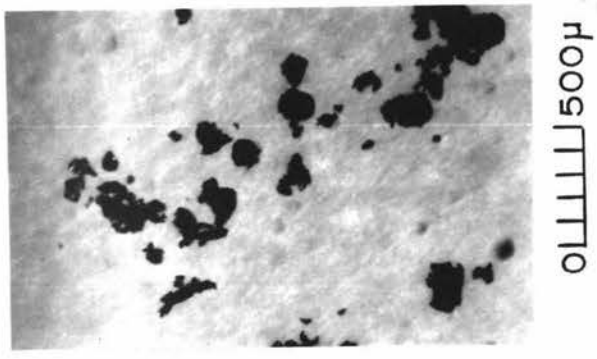
6. It should be mentioned that at a compression treatment of 50 psi no compaction effect (lumping) is evident and the compressed product is still truly a powder. At 150 psi some compaction is just evident but any lumps present break



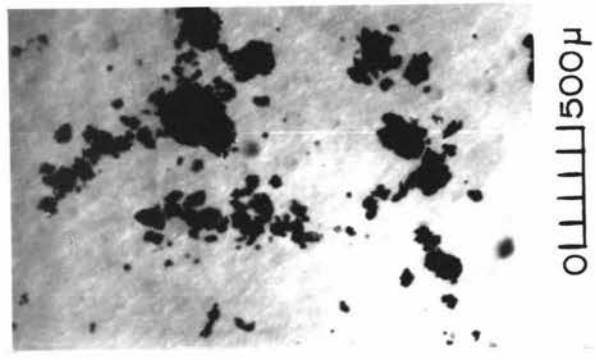
Control : 0 psi



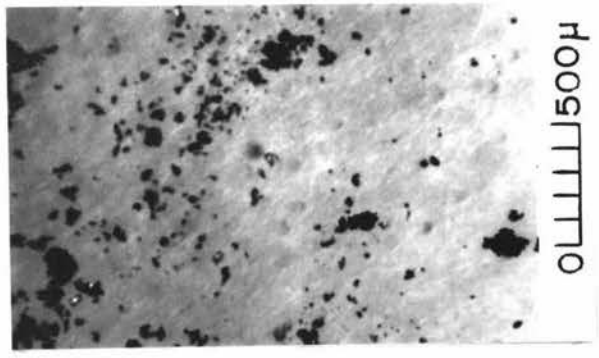
Compression : 16 psi



Compression : 102 psi



Compression : 282 psi



Compression : 1370 psi

FIGURE 15 Microphotographs Showing Effect of Compression/Repowdering Treatment upon Spray Dried Instant Coffee A (Magnification x 40)

up most readily;

7. Porosities of the reground powders are not shown in this case as they showed little variation, increasing only slightly on compression from approximately 0.63-0.67cc of voids per cc of loose powder. Porosities can be calculated from the particle densities and bulk densities shown in the graphs.

Some explanation of the phenomena observed can be made in terms of the working theory of dispersion :

The increase in dispersibility of powders compressed at low pressures can be attributed to an increase in the average particle density leading to a greater tendency for pacticles to separate from the powder/water interface once wetted. Eventually, however, higher pressures lead to a decrease in dispersibility which may be attributed to the high degree of fragmentation which has produced, virtually, a powder of much smaller particle size. As mentioned earlier, powders of finer particle size exhibit lower wettability and dispersibility characteristics.

Sinkability characteristics of the compressed powder are somewhat different from dispersibility. Initially there is evident a slight increase in sinkability which may be attributed to the increase in average particle density. However, until a pressure of approximately 350 psi is applied, average particle density is below 1.2gm/cc and it may be presumed that dispersion still occurs mainly at the powder/water interface. At the same time compression has the

effect of increasing the bulk density of the powder and this in itself may have the effect of decreasing sinkability while dispersion must occur from the interface. The influence of bulk density upon dispersion is supported by Harper et al (1963). They support and present evidence for the theory that concentration of solids in the vicinity of powder particles will have a marked influence on dispersibility. The bulk density of the powder will directly influence the concentration of solids in the vicinity of each particle upon reconstitution. Beyond 350 psi the sinkability of the powder again increases. By this stage the particle density has been increased beyond 1.2gm/cc and it may be assumed that dispersion no longer takes place at the interface only (i.e. Model 3). The limiting factor at these higher pressures now becomes the small particle size of the fragments and their resulting lower wettability (Pyne (1961)).

Although these explanations appear to satisfy the observed results they are tendered only from deductions which can be made. The process of reconstitution of a powder is obviously rather complex since many factors interact simultaneously to bring about dispersion. Nevertheless, some explanation has been possible by use of the proposed working theory of dispersion.

However, from a technological point of view, rather than theoretical, these results have more direct implications. The fact that spray dried coffee can be subjected to relatively mild compression without adverse effect on reconstitution characteristics immediately suggests certain commercial applications :

1. Results suggest the possibility of compressing coffee powders at low pressures, e.g. 150 psi, in order to achieve a higher bulk density product without adverse effect on dispersibility. Since little compaction occurs below 150 psi a "regrinding" operation may not be necessary. As pointed out above, treatment of 150 psi would increase bulk density of the repowdered product from 0.24 to 0.35gm/cc;
2. Results also suggest the possibility of compressing coffee powders, using low pressures, and packing the product at compressed volume. Obviously this would achieve even greater savings in packaging volume. Providing no compaction had occurred on storage the "compressed pack" could be transferred to a larger container by the consumer in order to release the repowdered product. Alternatively, the "compressed pack" could contain the exact quantity for a particular reconstitution;
3. Results indicate that compression has been used to modify the particle density and bulk density of spray dried coffee with no adverse effect upon dispersibility characteristics. This suggests the possibility of modifying spray drying conditions so as to produce a powder of higher bulk density and particle density. For example, high inlet air temperatures in spray drying tend to promote ballooning of particles, giving a powder of low particle density and bulk density, similar to spray dried coffee. It may well be that manufacturers have encouraged high inlet air temperatures

believing this to produce an instant coffee of optimum dispersibility. These results, at least, would justify some research on modification of drying technique in view of potential savings in bulk volume.

It is believed that the technological applications of this research show considerable potential and for this reason some follow-up work was done with regard to applications 1 and 2 above. Several points warranted investigation :

1. Since very little compaction occurs below 150 psi it would be interesting to compare dispersibilities of samples merely compressed and then transferred directly for reconstitution, versus samples compressed and then repowdered by use of the Moulin grinder and 850/u sieve as described in PROCEDURE;
2. If powders were to be compressed at low pressures and held at compressed volume a much greater saving in packaging would be possible as compared to repowdering, or "regrinding" before packaging. Determination of compressed bulk densities was warranted. Also, it appeared possible that some deterioration in dispersibility could occur if samples were stored at compressed volume; this warranted elucidation.

In this follow-up work 50gm samples of spray dried coffee A were compressed in standard 8oz cans using a specially made punch, or piston, which just fitted inside the can. With a little care powder could be compressed "in can" to over 150 psi without damage to the can. In two cases it was desired to store samples at compressed volume for four

weeks. This was done by placing a spacer into the remaining headspace of the can and then sealing the can. Coffee samples were given different treatments and then tested for dispersibility as presented in Table 3 below. Compressed bulk densities of coffee powders were determined by measuring the volume occupied by a known weight of powder in the compression die following a particular compression. These results are presented in Figure 16.

TABLE 3

Effect of Different Compression Treatments Upon Dispersibility of Spray Dried Coffee A

Treatment	Dispersibility %
Control Instant Coffee A	48.1
47 psi, not repowdered, not stored	51.3
47 psi, repowdered, not stored	49.9
47 psi, stored four weeks at compressed volume, repowdered	42.9
138 psi, not repowdered, not stored	53.8
138 psi, repowdered, not stored	48.6
138 psi, stored four weeks at compressed volume, repowdered	42.2

The following points are noted from the results :

1. Compression of instant coffee A up to 150 psi followed by repowdering of the product in the standard manner resulted in a slight increase in dispersibility;

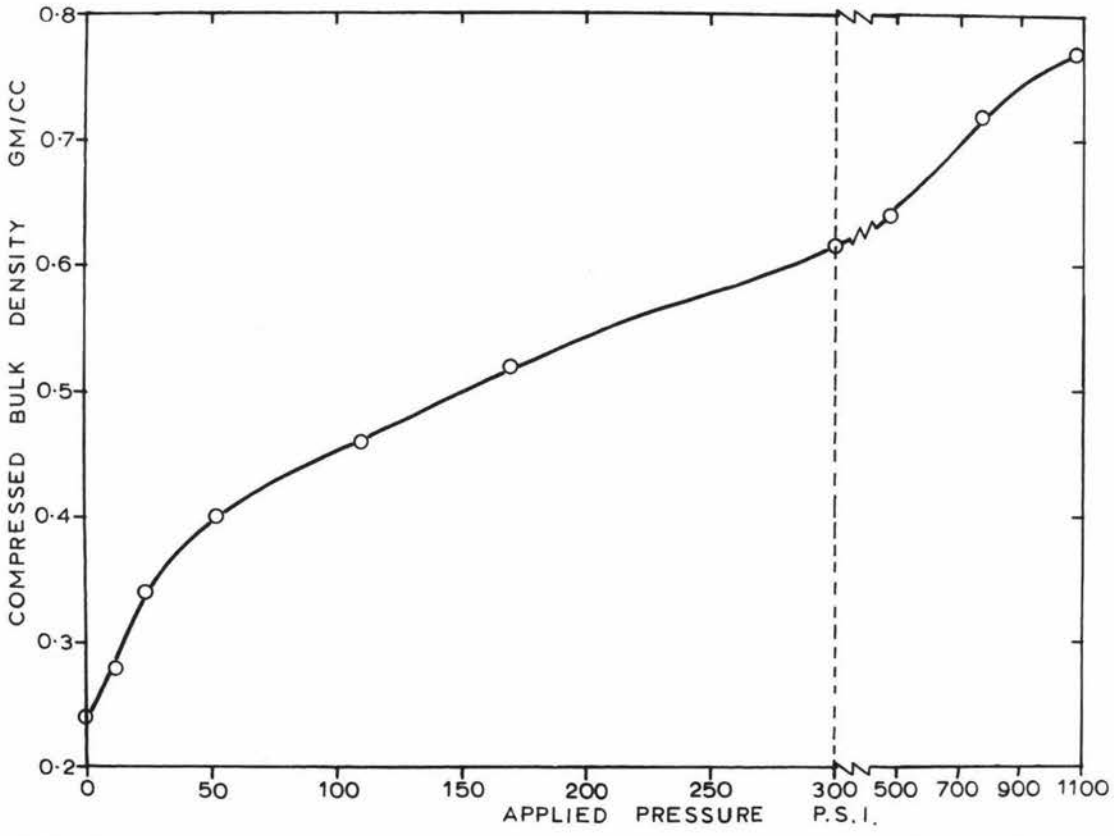


FIG. 16 COMPRESSED BULK DENSITIES OF INSTANT COFFEE A

2. At both pressures studied, 47 psi and 138 psi, insufficient compaction had occurred to affect the dispersibility of compressed powder not reground in the standard manner;

3. When Figure 16 is compared with the bulk densities of the repowdered coffee as shown in Figures 13 and 14 it is seen that an even greater saving in packaging space can be achieved if the powder is kept at compression volume. For example, at 150 psi treatment the compressed bulk density is 0.5gm/cc, representing a 52% saving in packaging volume over the original powder, compared with the 31% saving if repowdered;

4. Storage of treated samples at compressed volume for four weeks results in a slight decrease in the dispersibility of the repowdered coffee. The reason for this is not altogether clear. It may be that on holding at compressed volume, even though pressure is released vertically, internal stresses are retained. Such internal stresses could act in increasing the degree of fragmentation in the powder upon storage as illustrated in Figure 15. This could mean that the effect of storage is ,in fact, similar to the application of a higher pressure initially. More research on this aspect would be desirable before any conclusions are offered.

It is fully realised that the follow-up work which has been done with a view to technological applications of compression of spray dried coffee can serve as little more than a preliminary investigation.

Nevertheless, it is believed that results obtained are conclusive enough to justify further investigation. In addition such techniques of compression could have applications with other spray dried products. For example, sodium caseinate has a tendency to "balloon" considerably during spray drying, as do detergents, resulting in a product of low particle density and bulk density. As in instant coffee, therefore, the possibility exists of increasing bulk density and particle density of such powders with little if any adverse effect on reconstitution characteristics.

(ii) Temperature of Water for Reconstitution

The effect of temperature of reconstitution upon dispersibility of two samples of spray dried NFDM was determined. In all other work the temperature of the water in the dispersibility test is 75°F (23.9°C); in this case the temperature was varied and measured immediately before the placing of powder onto the water surface. Results are shown in Figure 17.

Both the Niro NFDM and the Modified Niro NFDM show a marked increase in dispersibility as the temperature of water for reconstitution is raised to 50°C. Above 50°C the Niro NFDM shows a sharp decrease in dispersibility from a maximum of 54% to approximately 40%. The behaviour of the Modified Niro NFDM is not as straight-forward, showing a decrease in dispersibility on raising temperature from 50°C to 60°C but showing another peak at 70°C. This result is almost identical to that obtained for wettability versus water temperature on a sample of spray dried NFDM by Gibson and Raithby (1954). They do not offer an

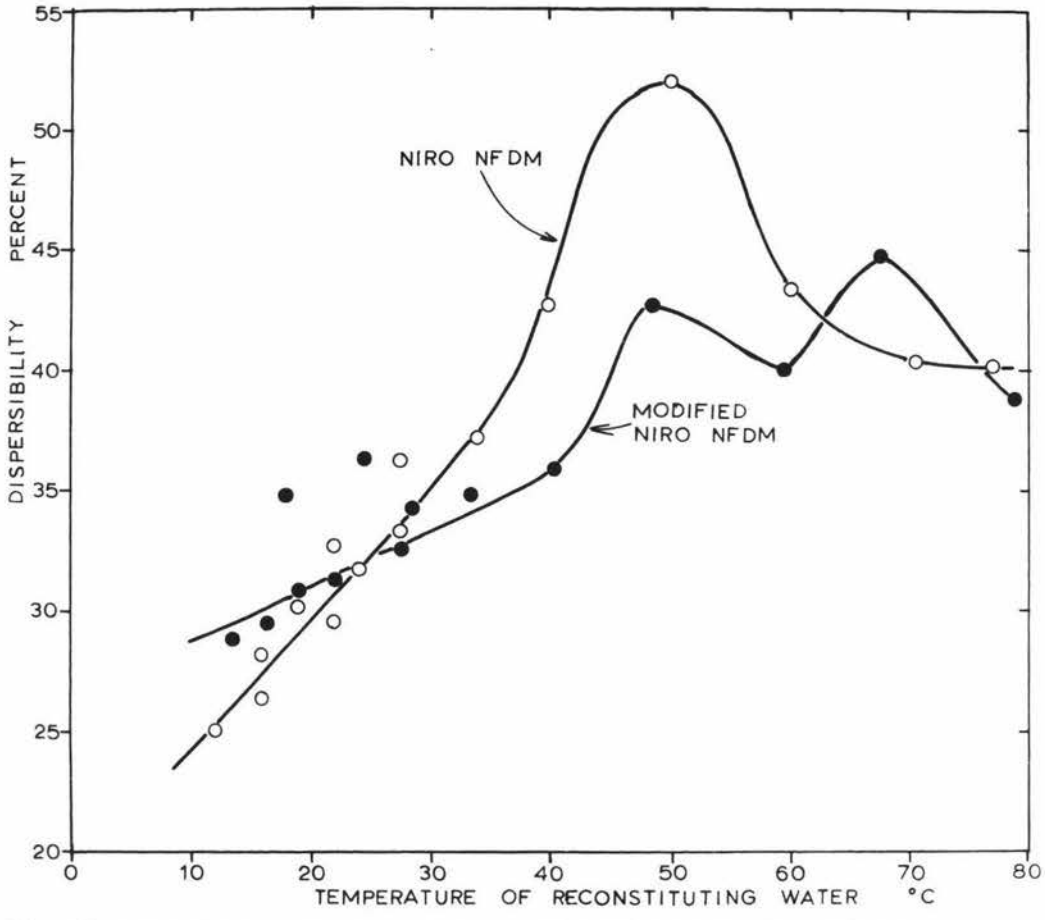


FIG.17 TEMPERATURE EFFECT ON DISPERSIBILITY OF NFDM

explanation for this observation.

As discussed in the literature review, the decrease in dispersibility above 50°C can be attributed to protein denaturation. This corresponds with work of Howat and Wright (1933).

It has been shown, therefore, that improvement in the dispersibility of NFDM can be achieved by a non-rewetting process, namely, by raising thetemperature of water for reconstitution to an optimum value. This optimum appears to be approximately 50°C for NFDM. The technique of determination could be used to determine optimum temperatures for other food powders. The improvement in dispersibility achieved for NFDM is quite considerable, e.g. from 25% at 12°C to 54% at 50°C for the Niro NFDM, particularly when this is compared with dispersibilities of approximately 60% for instant NFDMs at 24°C (Table 2). Results indicate that raising of reconstitution temperature becomes a compromise between an increasing dispersibility as against a decreasing net solubility, due to protein denaturation.

(iii) Addition of Free Flow Agents

The effect of addition of two free flow agents upon the dispersibility of Niro NFDM was studied. The addition of the agents to NFDM was carried out by dry mixing, employing two separate mixing steps, since concentrations of less than 5% were required. For example, free flow agent + NFDM to give a 10% mix was first blended thoroughly. This was then followed by blending of 10% mix and NFDM to give the required concentration of free flow agent in final mix.

Magnesium Stearate was added to Niro NFDM at the 2% level only. This work was done in conjunction with the study of effect of Stearate as a lubricant in compression of NFDM. The effect of Stearate addition upon dispersibility, sinkability index, and bulk density of the powder is summarised in Table 4.

TABLE 4

Effect of Magnesium Stearate Addition Upon Reconstitution of NFDM

Sample	Dispersibility %	Sinkability Index $mg/min.cm^2$	Bulk Density (Tapped)gm/cc
Niro NFDM	31.7	4.0	0.50
Niro NFDM + 2% Mag. Stearate	19.8	1.15	0.55

The other free flow agent studied was Syloid 244. This is one of a range of synthetic silica gels. Syloid 244 has an average particle size of 3 microns and even then each particle possesses a large internal porous structure. The bulk density of Syloid 244 was measured as 0.059gm/cc. The properties and applications of these synthetic silicas are described in technical literature by Grace (1966).

The effect of addition of Syloid 244 to Niro NFDM at concentrations of 0% to 5% was studied with reference to dispersibility and tapped bulk density. Results are shown graphically in Figure 18.

The following points may be noted from the results :

1. The addition of Magnesium Stearate to NFDM causes considerable

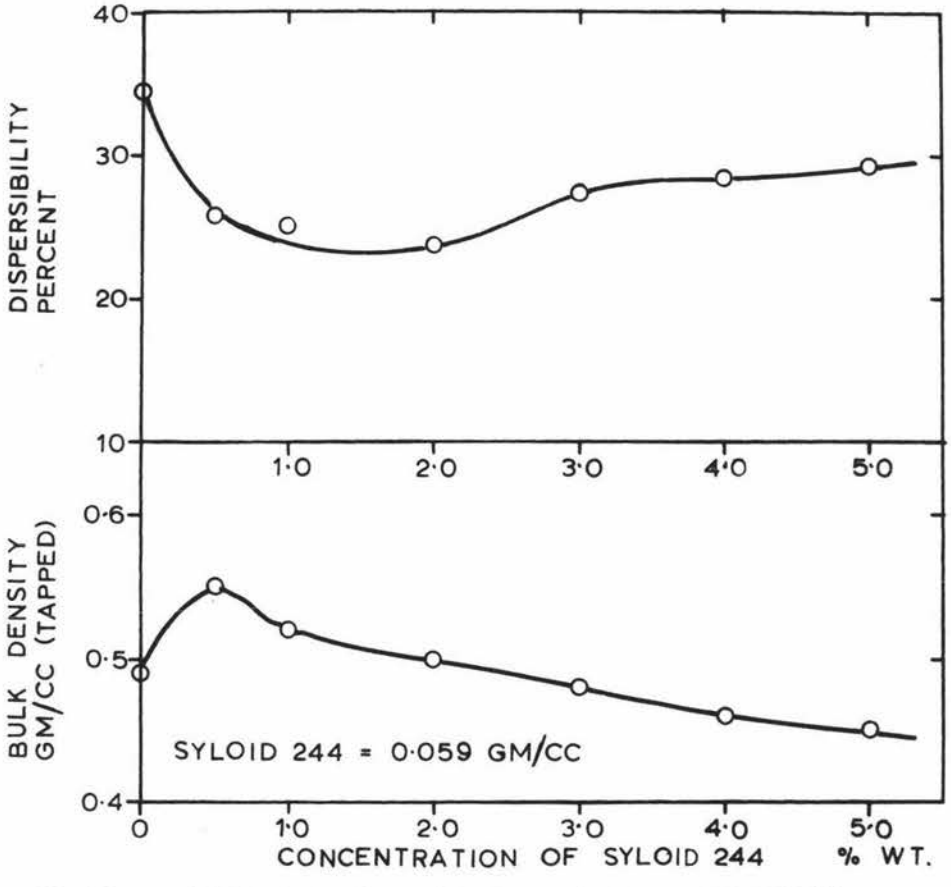


FIG. 18 EFFECT OF SYLOID 244 ADDITION TO NIRO NFDM

reduction in dispersibility. As mentioned previously this can be attributed to the hydrophobic character of Stearate, which in effect coats particles to produce a product similar to FCDM in reconstitution properties. This hydrophobic effect is reflected in the much lower sinkability index upon Stearate addition;

2. Syloid 244 addition causes a small decrease in dispersibility showing a minimum dispersibility at the 2% level, and increasing again on further addition;

3. Both free flow agents are able to increase bulk density of NFDM. Beyond 2% addition of Syloid 244, however, a reduction in bulk density is noted. This is to be expected in view of the extremely low bulk density of this agent (0.06gm/cc). Linton-Smith and Hansen (1963) also noted the reduction in bulk volume which could be achieved with free flow agents;

4. It is most interesting in Figure 18 to compare dispersibility with bulk density of the powder. Low concentrations of Syloid 244 bring about an increase in bulk density of the powder commensurate with a decrease in dispersibility. High concentrations of this agent decrease bulk density below the value of control powder while dispersibility now increases approaching that of the control. This observation therefore, suggests that the effect of Syloid 244 addition upon dispersibility may be largely due to modification of bulk density. This is supported by work of Harper et al

(1963) which demonstrated the effect of bulk density upon instant solubility of milk powders. They suggest that the lower the bulk density of a powder, the lower will be the concentration of milk solids in the vicinity of powder particles on reconstitution and hence the higher its instant solubility.

Both these flow conditioning agents when added to Niro NFDM produce a marked improvement in free flow properties as assessed subjectively. Even at 0.5% Syloid 244 addition the improvement is marked. However, it is shown that, for these two agents at least, this necessitates some sacrifice in the dispersibility of the powder. This may be a satisafactory compromise in some applications as, for example, in the case of milk powder for vending machines as described by Linton-Smith (1961). The hydrophobic nature of Stearate serves to explain the decrease in dispersibility in one case while with the silica some bulk density effect may be involved. However, silica gel also possesses strong water sorption properties suggesting that this agent may not be completely inert in the reconstitution process. It would be interesting to study the effect of a completely inert agent, if such exists, upon dispersibility of NFDM. Sodium aluminium silicate may possibly be such an inert free flow agent but a sample was unprocurable for this study.

E. PRINCIPAL RESEARCH FINDINGS

1. A study has been made of the effect of compression, followed by repowdering, upon physical properties and reconstitution characteristics of NFDM and spray dried instant coffee;
2. It has been shown that no significant increase in the dispersibility of NFDM can be achieved by such compression techniques; beyond a treatment of 1,500 psi there results a gradual decrease in dispersibility;
3. Compression treatment of both powders results in a marked increase in particle density. It is shown that much of this particle density increase can be attributed to fragmentation of the hollow spray dried shell;
4. Compression of both NFDM and instant coffee to particle densities of approximately 1.2gm/cc results in an increase in the sinkability index of the powder, even though such treatment affects adversely the dispersibility;
5. Compression of instant coffee up to 150 psi results in a slight improvement in dispersibility. Therefore, it is shown that, without adverse effect upon dispersibility, a treatment of 150 psi can effect a saving of 31% in packaging volume of the repowdered instant coffee or of 52% if packaged at compressed volume;
6. It is shown that 50°C appears to be the optimum water temperature for dispersion of NFDM. Above 50°C there is evident a net decrease in dispersibility attributable to increased protein denaturation;

7. It was found that addition of free flow agents to NFDM resulted in much improved flow and general handling properties although this improvement necessitates some compromise with dispersibility. Free flow agents may also effect an increase in the bulk density of a powder.

III. EXPERIMENTAL WORK

SECTION (3) EFFECT OF REWETTING PROCESSES UPON DISPERSIBILITY

SECTION (3) - EFFECT OF REWETTING PROCESSES UPON DISPERSIBILITY

A. INTRODUCTION

It is well known that NFDM and other food powders, such as chocolate mix, sugars, starches, etc. may be "instantised" by a rewetting/redrying process. Mori and Hedrick (1965) emphasise the importance of the instantising process in U.S.A. and state that practically all NFDM for home use in that country is now instantised. In 1963 this amounted to 255.3 million lb or 28.5% of total non-governmental use. Hall and Hedrick (1961) describe the manufacture of instant milk powders, while other agglomerated and instantised food powders are dealt with by Moore et al (1964); Claus and Brooks (1965); Miller and Powell (1965); and Lachmann (1966).

What is not so well recognised, however, is that such instantising processes are, in fact, little more than size enlargement or agglomeration operations commonly employed in many other industries. One such operation is that of wet granulation as practised in the pharmaceutical industry and in the fertiliser industry.

Undoubtedly, much work has been done on the agglomeration of food powders, and in particular NFDM, by instantising type processes. Unfortunately, however, very little such work has been published as most instantising processes are tightly controlled by patent rights. Nevertheless, the paucity of published information does suggest that little research has been carried out on the basic factors controlling the agglomeration process; probably due to the difficulty in setting up controlled experiments in commercially

available instantising equipment.

Keeping in mind that the basic steps in commercial instantising processes are no different to the basic steps in wet granulation of powders, it was the aim of this study to simulate an agglomeration process employing the principles of wet granulation. Once a simulated instantising process has been developed it can then be readily employed to study some of the basic factors controlling the process.

Research into rewetting processes is described under four headings, viz. :

(i) Effect of Rewetting Moisture;

(ii) Effect of Granule and Particle Size;

(iii) Uniformity of Moisture Distribution and Effect of Mixing during Granulation;

(iv) Additives in Wet Granulation.

B. LITERATURE REVIEW

(i) Effect of Rewetting Moisture

Mention is made in patents on instantising of milk powders of the range of rewetting moistures to be employed. Rewetting may be either by means of finely atomised water, steam, moist air, or a combination of these. The range of rewetting moistures specified in patents shows great variation and, in fact, forms the basis of discrimination between several patents. At the same time, it is realised in the industry that the extent of rewetting must be a

critical factor in producing a suitable agglomerated product, but no work or research is reported in the literature on this aspect. Probably the amount of rewetting employed in manufacture is a matter of subjective assessment by comparison with the final product.

Bullock (1962) in a review article discusses the manufacture of "Two-Stage" instants. Some of these methods for instantising may be listed :

1. Peebles was granted patents in 1955 and 1958. In this method powder is moistened by finely atomised water and air saturated with steam to a moisture content of 10-20% before redrying. In a description of the process, Peebles (1956) makes no mention of the moisture content of the rewet powder. However, in his patent, Peebles (1958) states an optimum rewetting moisture of 15%. This is not qualified other than by : "introduction of too much moisture results in too high a total moisture content for the material being delivered to the table feeder whereby the material tends to form a doughy mass rather than a fluffy stream of aggregates and cannot be dried to form a satisfactory product. An insufficient amount of moisture also causes the material delivered to be unsatisfactory".
2. The Cherry-Burrell system involves the agglomeration of particles with moist high humidity air moving at high velocity. Bullock (1962) reports a rewetting moisture of 6-10% for this system. Yet Carlson et al (1956) in

describing this process mention a rewetting moisture of 10-20%;

3. Louder and Hodson (1958) were granted a patent for a process involving rewetting with steam to not greater than 9.0% and preferably 5.5% moisture;
4. Scott (1959) holds a patent for a process involving wetting with a spray of water or milk to 10-14% moisture.

As can be seen, a wide range of rewetting moisture contents are specified in patents, although perhaps the range employed in practice may be much narrower. Mori and Hedrick (1965) studied the effect of some processing variables on dispersibility, moisture content and bulk density of instant milk powders. This is the only work of this type found in the literature, yet they make no mention of effect of rewetting moisture.

The amount of moisture to be added for granulation in the pharmaceutical industry is known to be critical but is largely subjective and a matter of experience. Such practice in wet granulation is described by Peck (1958) and Little and Mitchell (1949).

Some research has been done on the mechanism of formation of granules from moist powdered material, in particular moist sand, in a tumbling type drum mixer; Newitt and Conway-Jones (1958); and Capes and Danckwerts (1965).

Newitt and Conway-Jones (1958) describe three states of water in an assembly of spherical particles. At low moisture contents water is held in the granule as discrete rings at the points of

contact of the particles, i.e. the Pendular state. At somewhat higher moisture content the rings coalesce to form a continuous network of liquid interspersed with air, i.e. the Funicular state. At still higher moisture content the pore spaces of the granule become saturated to form the Capillary state. These workers postulated that initially the feed to a granulator consists of moist particles which have partly coalesced to loose aggregates held together by pendular bonds. Upon kneading action in the granulator the internal pore space in the aggregates is reduced; hence if sufficient water is present the pores may become saturated to form stable granules. They found granule formation in fine sand to be very dependent upon moisture content, an optimum value being approximately 68% v/v.

Capes and Danckwerts (1965) extended the work of Newitt and Conway-Jones, also employing the tumbling action in a rotating drum. Uniformly sized sands were granulated and comparison was made of granulating moistures and void volumes of the samples. Again it was possible to show the dependence of rate of growth of granules upon moisture content, but these workers showed that significant growth occurs only when liquid contents used are equal to between 90% and 110% of the amount required to fill the voids in a highly compacted sample. In other words in the granulation of sand it is necessary to achieve saturation of the voids in the aggregated particles with liquid before a stable system is reached.

(ii) Effect of Granule and Particle Size

The particle size of milk powders has long been appreciated

as an important factor in reconstitution characteristics but little conclusive evidence is presented in the literature.

Bockian et al (1957) investigated factors responsible for increased dispersibility of instant dry milks. They noted that in comparison with spray dried NFDM which consisted of particles less than 200 mesh (74/u), that instant powders are very large aggregates in the range of 80 to 20 mesh (180/u-840/u). They suggested that this may be one of the prime factors affecting the dispersibility of instant NFDM.

Similarly, Peebles (1958) considers particle size to be an important factor in obtaining an instant powder of desired characteristics. He believes it important that approximately 80% of the powder remains on a 200 mesh screen, that is, possessing a particle size greater than 74/u.

Swanson (1955) studied the effect of particle size on dispersibility of NFDM. Using an air elutriation technique he was able to fractionate powders into the different size ranges and test small samples obtained for dispersibility characteristics. He concluded that for NFDM "particles of 30-35/u in diameter seem to wet and dissolve most readily", particle sizes on either side showing reduced dispersibility. There appeared to be no correlation between particle size and dispersibility in the case of FCDM.

Gibson and Raithby (1954) examined powders of different average particle size and concluded that particle size influences the wettability of NFDM. Similarly, Baker and Bertok (1959) graded NFDMs

for particle size by the use of sieves and examined the size fraction. They concluded that with a decrease in particle size there occurred a corresponding decrease in wettability and dispersibility.

Hall and Hedrick (1961) claim that manufacturers of instant NFDM are fully aware of the importance of particle size. They describe a desirable particle size distribution as being in the range 100-500/u; no more than 10-15% of weight of particles should be less than 150/u in size.

Mori and Hedrick (1965) studied the effect of certain processing conditions on the properties of instant milk powders. They found that dispersibilities were not consistently affected by particle size (tested on fractions of sieving).

Claus and Brooks (1965) describe some properties of instantised wheat flours. Normal wheat flour is required to pass a 100 mesh U.S. sieve (150/u), whereas these modified flours are granulated or instantised, to produce a much larger "particle" size. Figures are given for size distributions of instantised flours together with evidence of the much improved wettability, or sinking time, of these flours over the normal product.

It can be seen from a review of the literature that many researchers have illustrated the importance of particle size as a factor in dispersion of a food powder. Results, however, are far from conclusive and there appears to have been no satisfactory elucidation of optimum particle size, even for the case of NFDM. Pyne and Coulter (1960) emphasise that there must exist an optimum

particle size corresponding to optimum 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 the surface to mass ratio. It is reasonable to deduce, therefore, that an optimum surface to mass ratio, or particle size, exists corresponding to optimum reconstitution characteristics.

(iii) Uniformity of Moisture Distribution and Effect of Mixing during Granulation

Little information is available in the literature on the uniformity of moisture distribution on rewetting of a base powder. In commercial instantising processes this factor will be largely a function of agglomerator design and method of rewetting. As is revealed in the review by Bullock (1962), many different methods of rewetting are employed, including finely atomised water, steam and humidified air.

The uniformity achieved will also be influenced by the degree of mixing occurring during agglomeration, or granulation. Again, in commercial processes this will be largely a function of instantiser design. In wet granulation as practised in the pharmaceutical industry some control is possible as determined by the time of batch mixing employed. For example, Little and Mitchell (1949) state that for a simple formula approximately 15 minutes mixing time is required.

Apart from an effect on uniformity of moisture distribution, mixing of the powder may well have an effect on actual granule formation. This is suggested from the papers of Newitt and Conway-Jones (1958), and Capes and Danckwerts (1965). These workers suggest that a stable

granule, in the case of sand, is not formed until a capillary state of water is reached where saturation of the voids of the agglomerates is satisfied. In this regard mixing of a powder may achieve some degree of compaction, thus reducing voidage and making possible the formation of stable granules at lower moisture contents.

In the present study the main interest in these factors of moisture distribution concerns the technique of wet granulation employed to simulate the instantising process. It is desired to ensure that the methods of rewetting and mixing employed are satisfactory and adequate.

(iv) Additives in Wet Granulation

The use of various additives in wet granulation is most common in the pharmaceutical industry. Their application is described by Peck (1958); Little and Mitchell (1949); and Donaghy (1958). Such additives may be classed into three categories :

1. Binders. These are virtually adhesives which permit a heterogeneous powder to be formed into a granule. They are usually added in solution, some examples being gums, dextrin, gelatin, starch, lactose, and sugar syrups;
2. Disintegrants. These serve to promote capillary penetration of water into a tablet upon reconstitution. The most common disintegrant is starch;
3. Lubricants. Additives such as talc and stearate are employed to improve flow properties and to impart lubricating action during compression of granules.

Very little extension of the application of additives in wet granulation has been made to the granulation of food powders such as in instantising of NFDM. One of the main reasons for this is probably the difficulty in experimentation with commercial scale equipment.

However, the potential benefits to be gained by the use of additives in agglomeration has also stimulated Moore et al (1964). They suggest that where necessary additives may be used to achieve agglomeration of food powders in commercial instantising equipment. Such additives they suggest may be put into two categories :

1. Where acting as a binding agent. An example is given of the addition of sugar to cocoa powder, to the extent of 80% sugar, so as to act as a binder in agglomeration to produce an instant cocoa mix, or drinking chocolate;
2. Where an additive acts as an emulsifying agent or surface active agent to allow wetting of the particle surface.

The addition of surface active agents to milk powders in an attempt to improve dispersibility has been studied by many workers : King (1966); Nelson and Winder (1963); Gibson and Raithby (1958); Mather and Hollender (1955); and Hollender (1952). In all these cases the surface active agent was added to the milk or concentrate before drying. No report appears in the literature of surfactant addition on rewetting in agglomeration. However, it may be mentioned that surfactants form an important constituent of coffee whiteners as manufactured in U.S.A., being employed at levels of 0.3-0.5% dry basis.

Coffee whiteners are described by Hedrick and Armitage (1964) and by Atlas Chemical Industries Inc. (1965).

C. PROCEDURE FOR GRANULATION

Peck (1958) describes wet granulation as employed in the pharmaceutical industry as consisting of the following operations :

1. Mixing the fine powders with a liquid or solution which has or produces the required adhesion;
2. Forcing the moistened materials through a screen of suitable size mesh to form granules;
3. Drying the moist granules;
4. Screening the dried granules to produce the final and required size.

These four basic operations are exactly the same as those employed in this study for the manufacture of NFDM granules. Use was made of a Kenwood Chef mixer for the first two steps. This model Kenwood mixer employs "planetary" mixing ensuring efficient action. The "K" beater was employed for the rewetting operation, while the colander and sieve attachment was employed for forcing moistened particles through a screen. This latter attachment consists of a rotating paddle fitted with scraper blades which travel over the surface of a screen fitted inside the colander bowl. For most of this study the finer of the two standard screens was employed having holes of 1600/u diameter. It was also found necessary to control the speed of the mixer by means of a "Variac", variable voltage control

as the variable control provided with the Kenwood could not be regulated to the low speeds desired.

The four steps, then, as employed in this procedure are as follows :

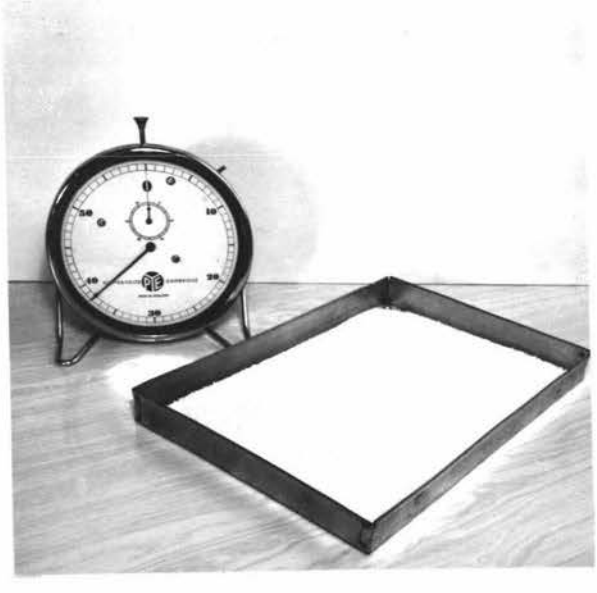
1. Two hundred gm of powder is placed in the mixer bowl and the "K" beater is set in motion. The quantity of water to be added is allowed to drip in slowly and in stages. Regular manual stirring using a rubber plate scraper is also employed so as to overcome any possible "dead spots" in the mixer. This mixing stage should take approximately 10-15 minutes;
2. After allowing the moistened mix to stand for 5-10 minutes granulation is carried out in the colander and sieve attachement, using the 1600/u screen;
3. The moist granules are spread onto a tray and air dried at 140°F in a fan assisted drier. Drying time is adjusted to give a final product of 3.0-4.0% moisture. The granules on the drying tray are stirred periodically during the drying cycle;
4. The dried granules are forced through a 2,000/u (B.S. 8 mesh) sieve. This is for the sake of uniformity, only, so as to eliminate any large clumps which may have formed during drying.

The stages in this granulating procedure are depicted in Figure 19.

In drying of moist granules it is desired to effect a minimum of heat damage. Some experimentation was, therefore, conducted to

(1) Mixing and Rewetting of Base Powder

(2) Granulation in Colander and Sieve Attachment



(3) Tray Drying at 140°F

(4) Dry Screening Through 2000/u Sieve

FIGURE 19 The Four Steps Employed in the Granulation Procedure

determine a satisfactory drying temperature/time combination which would give minimum damage. The solubility index of the dried granules was employed as an index of heat damage. Results are presented in Table 5.

TABLE 5

Effect of Various Drying Conditions on Solubility Index of Granules

Drying Conditions on Moist Granules	Initial Moisture	Final Moisture	Solubility Index
Control Niro NFDM	3.5%	-	0.10ml
110°F/165 minutes	11.5	4.0%	0.10
120°F/100 minutes	11.5	3.7	0.10
130°F/100 minutes	11.5	3.3	0.15
140°F/ 55 minutes	11.5	3.2	0.10
160°F/ 35 minutes	11.5	3.2	0.40

As can be seen, for granules initially at 11.5% moisture no heat damage is reflected in the solubility index until drying temperature is raised above 140°F. It was therefore, decided to tray dry all granules at 140°F for approximately 50-60 minutes, i.e. to reduce moistures to 3.0-4.0%.

D. RESULTS AND DISCUSSION

(i) Effect of Rewetting Moisture

As was discussed in the literature review there is considerable evidence to suggest the importance of rewetting moisture in determining stable granule or agglomerate formation. But, there is no report of the effect of rewetting moisture upon the properties of instant powders, as for example instant NFDM. Employing the technique of wet granulation described it is possible to study such relationships.

Two samples of NFDM, viz. Niro NFDM and Rogers NFDM, were granulated over a range of rewetting moistures. All these samples were granulated through the 1600/u screen and then dried and sieved as described in PROCEDURE. Rewetting moistures were determined from sampling of the moist granulated product not from the moistened mix. Some drying does occur during granulation, i.e. as moist powder is forced through the screen but this is not appreciable and it was found more satisfactory to sample after granulation.

Therefore samples for both NFDMs were granulated corresponding to a range of rewetting moistures. These were analysed for dispersibility, sinkability index, solubility index, bulk density and porosity. These results are presented in Figures 20 and 21.

Photographs of samples obtained in the granulation of Niro NFDM are presented in Figure 22.

Points arising from these results are best enumerated :

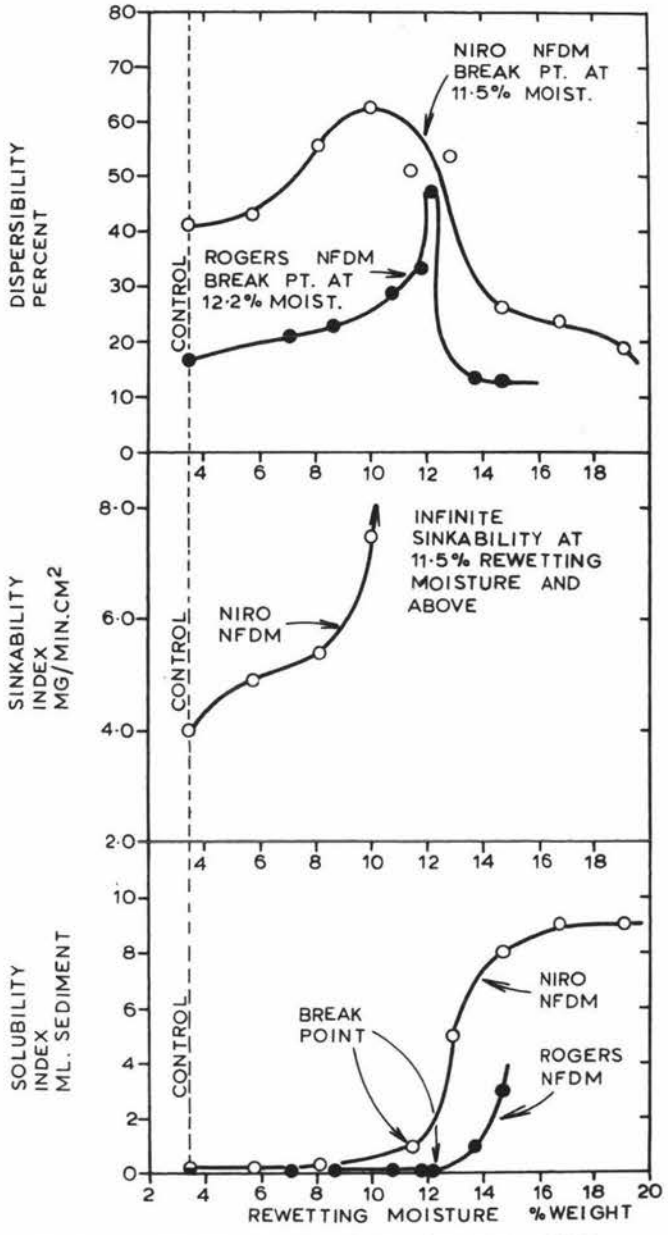


FIG. 20 PROPERTIES OF GRANULATED NFDMs (REDRIED)

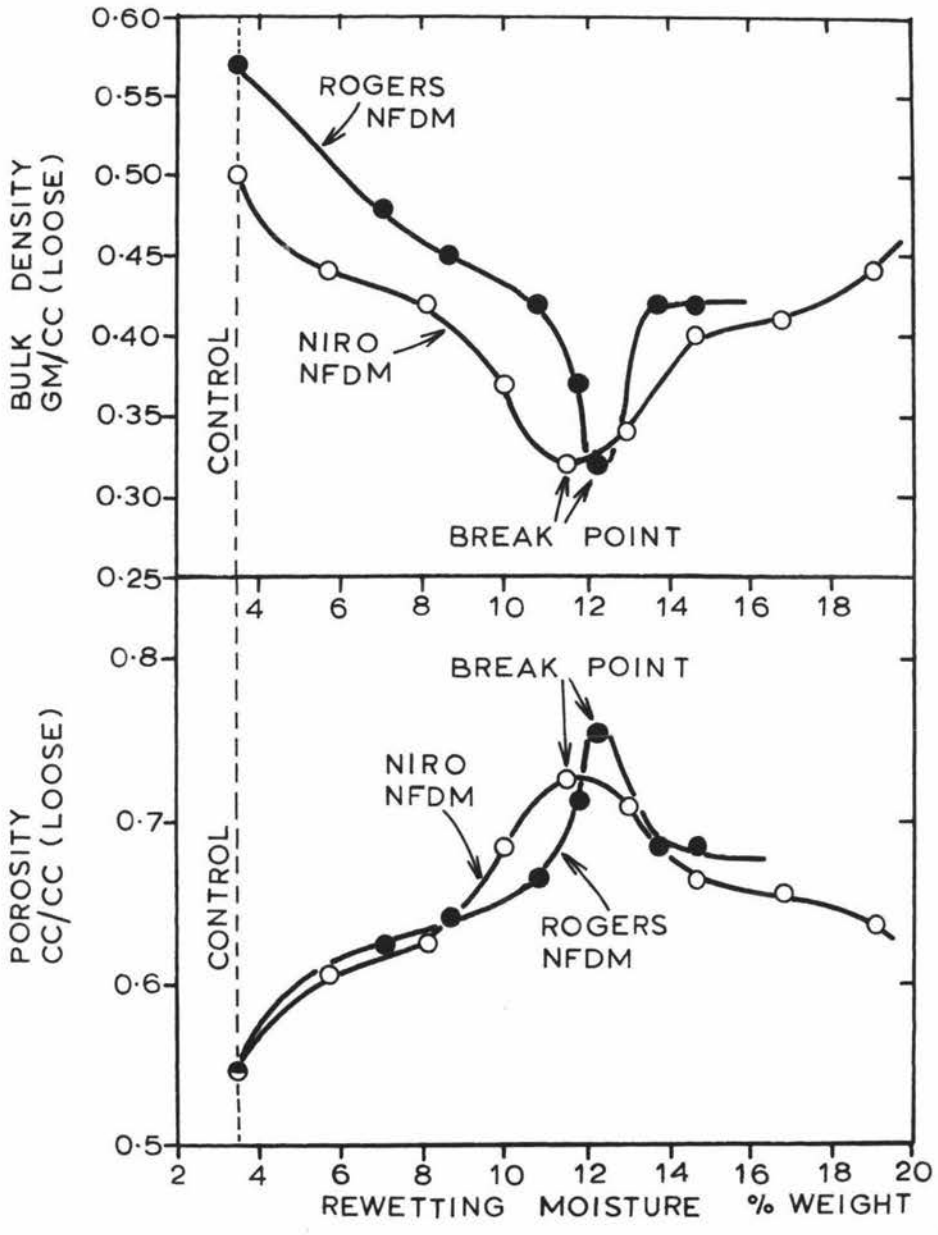


FIG. 21 PROPERTIES OF GRANULATED NFDMs (REDRIED)

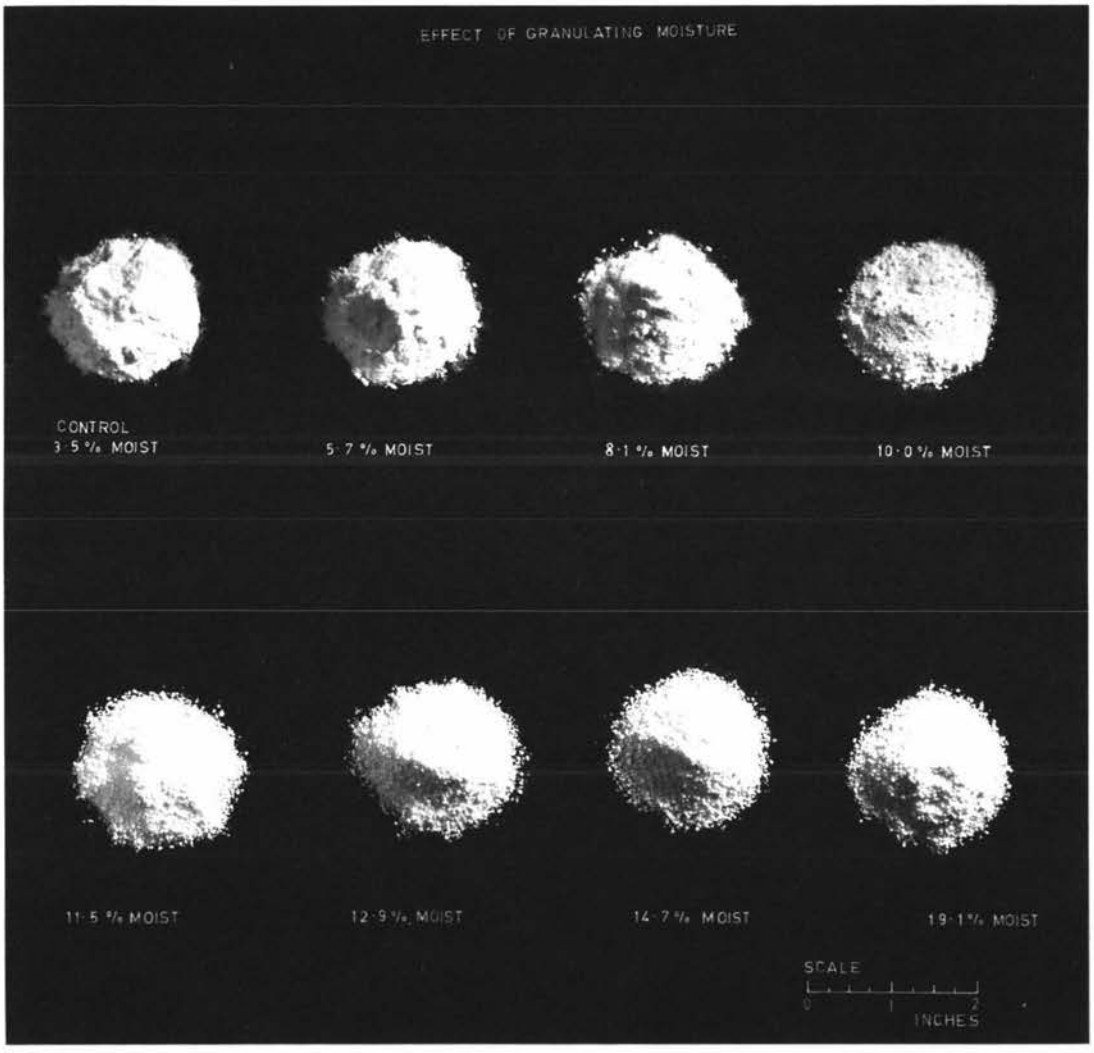


FIGURE 22 Samples of Redried Granulated NFDM Showing Effect of Rewetting Moisture Upon Granule Formation

1. The critical nature of rewetting moisture upon the properties of the final product is almost self-evident in every respect. Firstly, it was found that the rewetting moisture content controls the actual granulating process. As the moisture content is increased a point is reached where the "granulated" product changes from a fluffy type powder with poor flow characteristics to well formed granules of excellent flow properties. This change is quite marked as may be evidenced from Figure 22; the point at which this change occurs has been designated the BREAK POINT;
2. The effect of rewetting moisture upon dispersibility can be seen from Figure 20. The dispersibility of both NFDMs could be increased markedly and at optimum rewetting could be classed an instant NFDM (cf Table 2). The optimum dispersibility coincides with the Break Point rewetting moisture; a sharp decrease in dispersibility occurring as rewetting is increased further;
3. The sinkability index of the Niro NFDM is seen to increase with rewetting moisture, reaching an infinite value at 11.5% moisture; i.e. coinciding again with the Break Point. "Infinite Sinkability Index" is a relative term only since obviously some time is required for disappearance from the water surface, but in this case sinking is so rapid in comparison to sub-Break Point powders that it is termed infinite.

Although sinkability index values were not determined for Rogers NFDM samples they showed an identical trend when examined qualitatively;

4. Beyond Break Point moistures the solubility index of the samples is seen to show a sharp increase (i.e. a decrease in solubility). Below Break Point moistures the solubility index values are quite satisfactory and would pass A.D.M.I. tentative standards of 1.0ml;

5. The bulk density and porosity measurements shown in Figure 21 are quite interesting particularly when compared with the corresponding dispersibility curves in Figure 20. Again it is seen that minimum bulk density values, or maximum porosities, coincide with Break Point rewetting moistures. In fact, it is seen that there exists a strong correlation between dispersibilities and porosities of the samples;

6. Samples of the granulated Niro NFDM were also examined for state of lactose. It was found that as rewetting moisture was increased there occurred a gradual increase in the incidence of crystalline lactose present, with a sharp change to predominantly crystalline lactose coinciding again with the Break Point moisture. Actually, it was at this stage that it was discovered that crystalline lactose was present in the original Niro NFDM. This led to the follow up work previously reported; Morris, Neff and Latimer (1967).

It is interesting to compare the Break Point moistures obtained in the granulation of NFDM with those obtained for the

granulation of sand by Newitt and Conway-Jones (1958) and Capes and Danckwerts (1965). This comparison is made in Table 6.

TABLE 6

Comparison of Break Point Moistures in the Granulation of Sand and NFDM

	Sand	NFDM
Density : gm/cc	2.61	1.20
Void Volume : $\frac{\text{cc void}}{\text{gm solid}}$	0.270	0.83 (= 0.50cc/cc)
Granulation Break Point at :		
v/v moisture	68%	16.4%
cc/gm moisture	0.260	0.136
Void saturation	96%	16%

Using a Break Point value of 12% moisture for NFDM, this works out to 16.4% on a v/v basis, and to 16% void saturation as compared with 96% saturation in the granulation of sand. This illustrates clearly that void saturation is not necessary in the granulation of a non-inert powder such as NFDM; adhesion of particles in this case is no doubt assisted by dissolving solids which form a binder solution.

The increase in dispersibility and sinkability index up to the Break Point suggests that this is due largely to the process of size enlargement. This aspect is to be examined more closely with regard to studies on effect of particle size so will not be discussed

further at this stage. However, the decrease in dispersibility beyond the Break Point deserves some mention. It is seen that this corresponds with a sharp increase in the solubility index (i.e. a decrease in solubility proper). Further, it was observed that beyond Break Point rewetting samples of NFDM granules appeared definitely more yellow in colour than "powder" samples below Break Point. Much of this difference in colour could be attributed to an optical effect due to the increased particle size of granules, (this can be demonstrated by granding up granules to a finer particle size) but it was concluded that some of this yellow colouration, at least, could be attributed to a chemical change in the NFDM. Yet another observation was that at approximately 14% rewetting moisture the NFDM passed through a "doughy" stage during mixing. This was almost as if a gel state had been formed.

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 now 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 insolubilisation, in a NFDM system. Such changes are comprehensively discussed by Coulter et al (1951). It is noted that moisture is an important factor in determining rate of browning, the

optimum moisture in a NFDM system being approximately 12-14%.

An interesting point emerges with regard to the curves for Rogers NFDM as seen in Figures 20 and 21, viz. the very critical nature of rewetting moistures over a narrow range. The dispersibility characteristics of Rogers NFDM are modified over a much narrower moisture range than Niro NFDM, and similarly the bulk density and porosity curves are much steeper about the Break Point moisture. This observation serves to illustrate quite satisfactorily the notion held in the dried milk industry in U.S.A. that Rogers NFDM is most difficult to instantise. The reason for this is not clear but it may be noted from Table 2 that this NFDM possesses by far the lowest mean particle size, being less than 10/u.

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 (1963). 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 is most important in this regard also.

Peebles (1958) in his patent on instantising of NFDM notes that optimum rewetting, in his case claimed to be 15% moisture,

corresponds with a minimum bulk density of the product. He states : "If too much water is being introduced into the process it becomes readily apparent by a decrease in the apparent bulk of the material". Peebles stated this in the nature of a qualitative observation whereas the present study has shown conclusively the critical nature of rewetting moisture upon both dispersibility characteristics and bulk density of the product.

(ii) Effect of Granule and Particle Size

Up to this stage all granulation has been carried out using the 1600/u screen as described in PROCEDURE. This does not mean that all samples produced possess a particle size of 1600/u. Below Break Point rewetting true granulation does not occur and although some agglomeration takes place the powder will have a mean particle size considerably below 1600/u. At Break Point rewetting true granulation is just occurring and the powder will exhibit a wide size distribution. Above Break Point rewetting the size distribution again narrows and the mean particle size approaches that of the true granules; even the size of these granules, however, will be below 1600/u since syneresis of particles occurs during drying.

Now from a literature review it is to be expected that particle size would show considerable influence upon reconstitution characteristics. This is further supported from the results showing effect of rewetting moisture upon reconstitution, where it was found that optimum dispersibility was promoted at Break Point rewetting. But the interesting point to note here is that at Break Point rewetting

a wide size distribution is to be expected. This emphasises the desirability of determining optimum particle size of NFDM with respect to dispersibility characteristics.

Determination of optimum particle size was approached in three ways :

(a) Granulation through different size screens at above Break Point moisture;

(b) Sieving of large sample produced at Break Point rewetting to give different size fractions;

(c) Grinding of large sample of granules produced at above Break Point rewetting followed by sieving to obtain different size fractions.

(a) Granulation Through Different Size Screens

The smallest screen supplied with the Kenwood Chef has holes of 1600/u diameter. In order to produce granules of different sizes special screens were made to fit the Kenwood attachment by soldering standard wire mesh onto supporting perforated base plates. Batches of Niro NFDM were then moistened as described in PROCEDURE using the same quantity of rewetting water in each case so as to yield granules of approximately 14% moisture; in this case, however, granulation was achieved through the specially manufactured screens.

Although screens down to 80 mesh were made it was found that 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 is decreased. This is evidenced in the drying which occurs during actual granulation - increasing difficulty in granulation leading to greater moisture loss. This can be seen from Table 7 showing the moisture content of the granulated samples. All batches were rewet with equal quantities of water.

TABLE 7

Moisture Content of NFDM Granulated Through Different Size Screens

Screen Size	Aperture Microns	Moisture %
10 mesh	2000	14.0
Kenwood screen	1600	13.6
16 mesh	1000	13.7
30 mesh	500	13.3
40 mesh	390	12.7

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. Rewetting was well above the Break Point and good granules were formed in all cases.

Samples were analysed for dispersibility, porosity and solubility index. These results are presented in Figure 23.

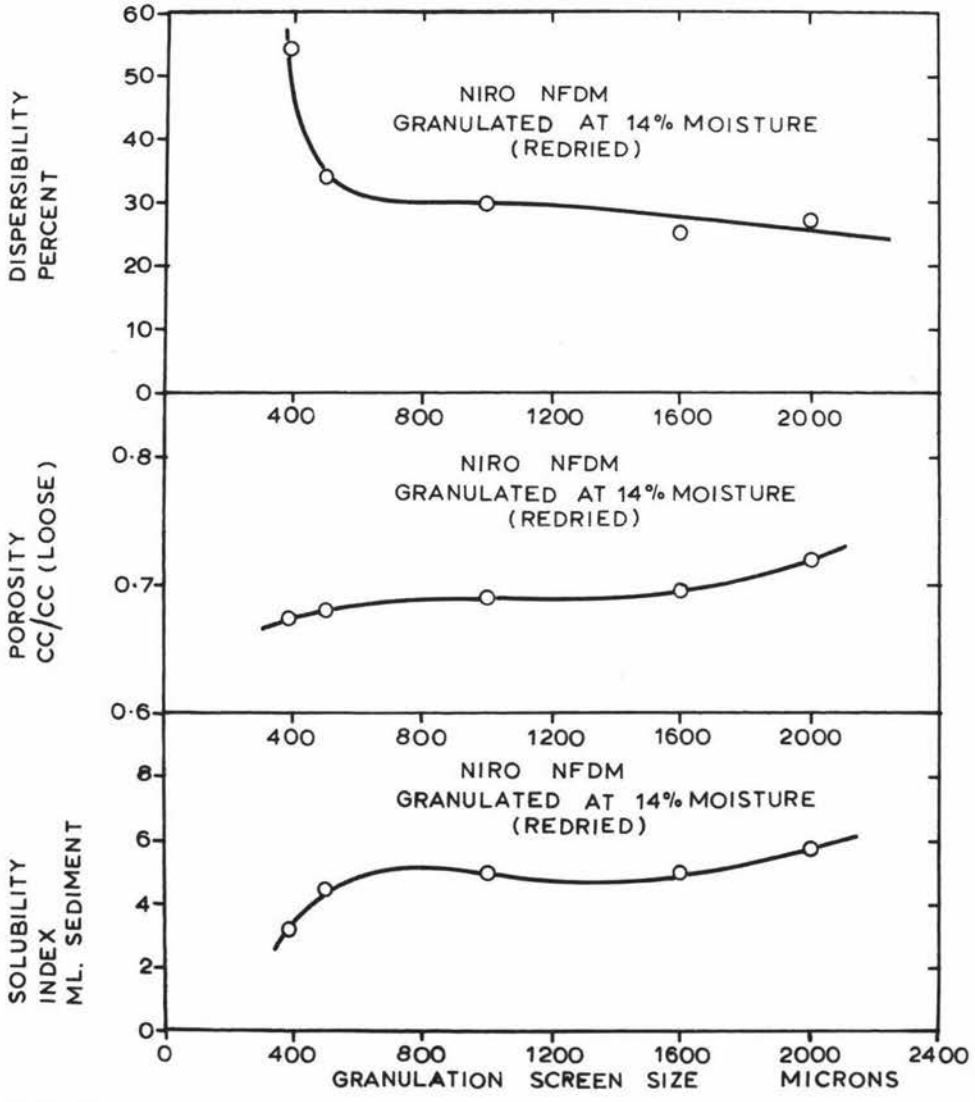


FIG. 23 EFFECT OF GRANULATION SCREEN SIZE UPON PROPERTIES OF NFDM

(b) Sieving of Samples Produced at Break Point

Four batches of Niro NFDM were granulated as described in PROCEDURE using the 1600/u screen. The same quantity of moisture was added to each batch so as to correspond to approximately Break Point rewetting. The moisture content of the moist granules was determined to be 11.6%.

As mentioned previously at approximately Break Point rewetting, 11.6% in this case, a wide size distribution of particles is to be expected. Production of 4 batches therefore, allowed sieving of the dried product to yield sufficient fractions in each size range for analysis. Sieving was carried out using B.S. test sieves together with a mechanical sieve vibrator. All powder handling and sieving was carried out in a R.H. controlled room held at 40% R.H. so as to minimise moisture pickup by the powder and facilitate handling, etc.

Eight sieve fractions were obtained by this technique. These were analysed for dispersibility, and sinkability index, the results being presented in Figure 24. In addition a particle size distribution curve was constructed from the results of sieving, this being presented in Figure 25.

(c) Sieving of Ground Granules

Four batches of Niro NFDM were granulated as described in PROCEDURE, using the 1600/u screen. Again the same quantity of moisture was added to each batch but this time this corresponded to 14% moisture in the 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 type grinder, gave a wide size distribution

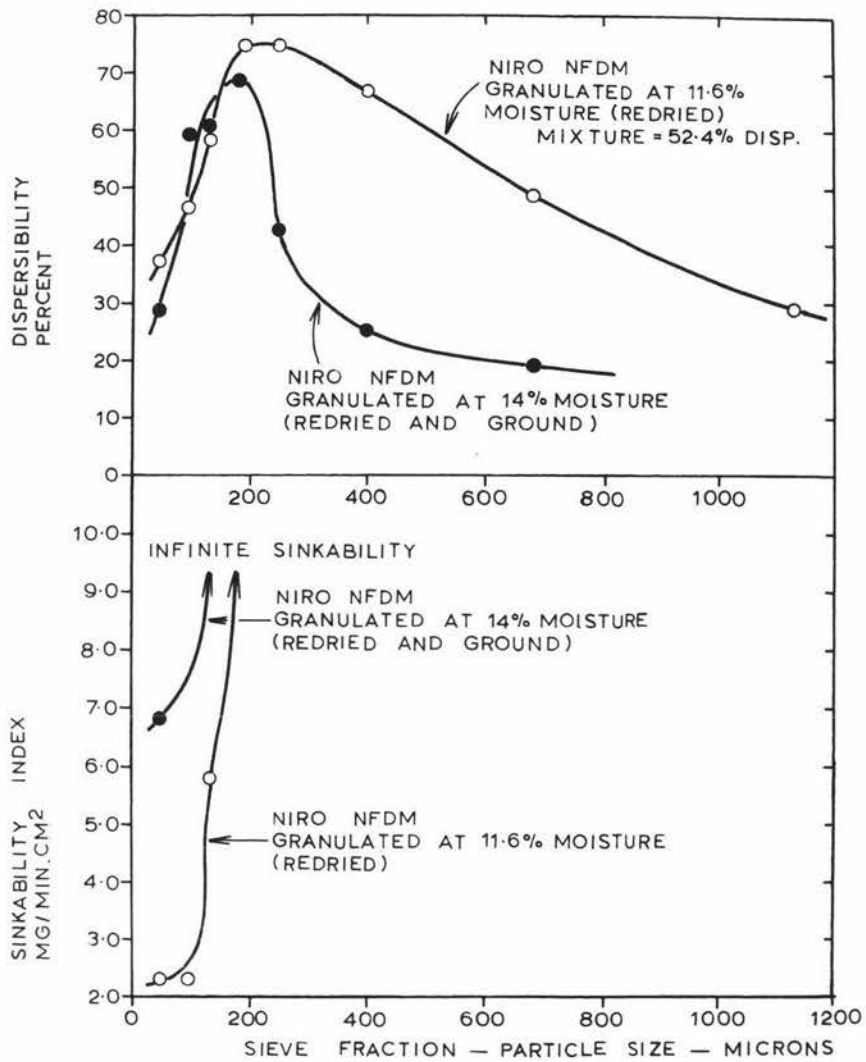


FIG. 24 EFFECT OF PARTICLE SIZE UPON RECONSTITUTION OF NFDM

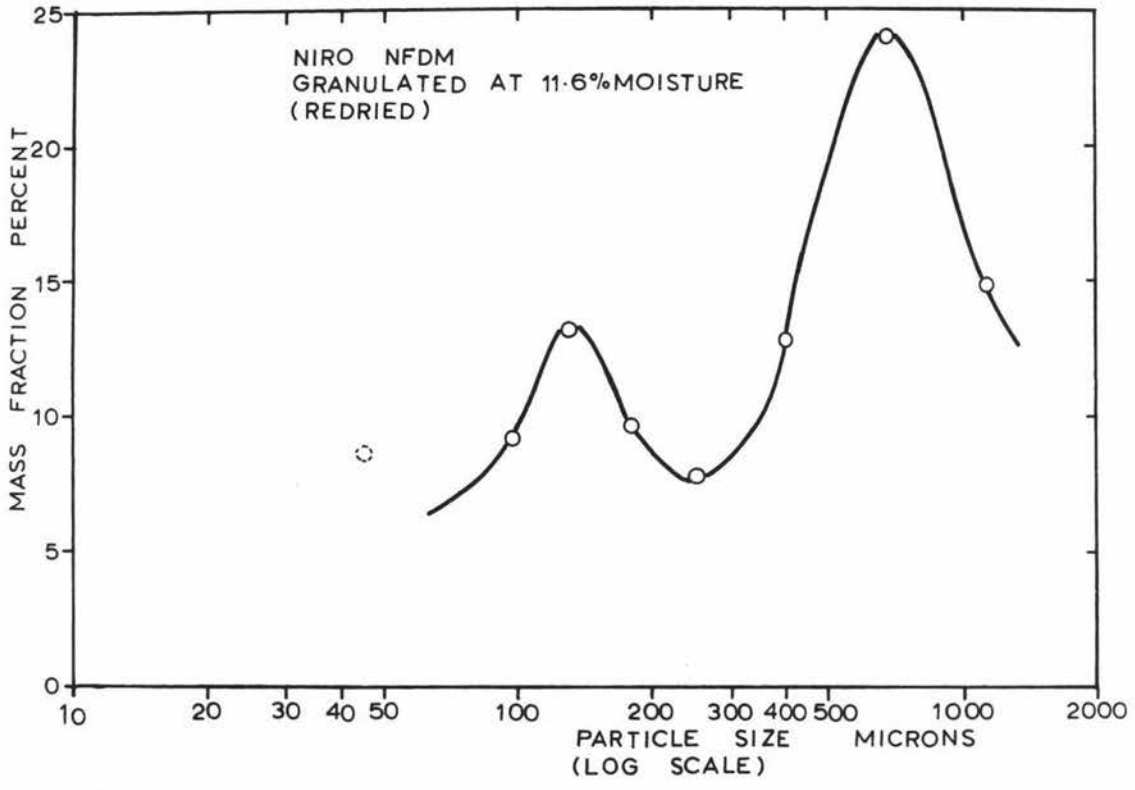


FIG. 25 PARTICLE SIZE DISTRIBUTION OF BREAK POINT GRANULATION OF NFDM

suitable for this purpose.

As before the ground granules were sieve separated to give 8 fractions, sieving and handling being done in a 40% R.H. controlled room. The separated fractions were analysed for dispersibility and sinkability index, these results being presented in Figure 24.

Points arising from the results in Figures 23,24 and 25, may now be discussed :

1. From Figure 23 it may be seen that dispersibility increases as the granulation size is decreased from 2000-390/u. It must be remembered that this curve represents granules produced at 14% rewetting which, as may be seen from Figure 20, is well beyond the optimum rewetting moisture. Yet even so the dispersibility is increased from 25.0% to 54.3% merely by decreasing granule size. As wet granulation could not be achieved through finer screens an optimum size for granulation was not determined, i.e. all that can be deduced is that an optimum granulation size will be less than 390/u;
2. Porosity and solubility index determinations in Figure 23 show only very slight change with granulation size. Theoretically, porosity will be independent of particle size providing a population consists of spherical particles all of the same size. This correlates well with the porosity determinations, the decrease in porosity with decrease in particle size being explained by a relative increase in particle size distribution. The slight increase in solubility with decrease

in particle size also appears reasonable, in view of the greater surface/mass ratio of small particles;

3. Figure 24 shows the effect of particle size upon reconstitution characteristics for a more complete size range. In this case an optimum particle size has been determined. In both granulated NFDMs, i.e. rewetting to 11.6% and to 14%, the optimum particle size as determined by sieving is approximately 200/u. A particle size of 200/u represents optimum dispersibility together with infinite sinkability. It may be emphasised that the maximum dispersibility of 75% obtained for the optimum size, optimum rewetting, fraction is the highest obtained for any instant powder in this study including several commercial instant NFDMs;

4. It is noted that beyond 130/u and 180/u particle size for the 14% and 11.6% rewet granules respectively there is established an infinite sinkability index, i.e. powder no longer "floats" on the surface of reconstituting liquid. The lower size at which this occurs for the 14% rewet granules may be attributed to their higher particle density, although no particle density measurements were made in this instance;

5. An attempt was made to establish a theoretical model to account for the sudden change from low to infinite sinkability of a particle as its size is increased. This proved to be a rather complex problem which, if it was to be treated quantitatively, required a knowledge of solid/liquid surface tensions and contact angles at the water surface. From a qualitative point

of view, however, this phenomena is to be expected. This 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 R^3. 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 as 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/u for the NFDM granules studied.

6. From the particle size distribution curve in Figure 25 it is seen that at Break Point rewetting the final product consists of a population with two definite peaks at 130/u and 680/u. This is interesting in view of the fact that optimum size is approximately 200/u; indicating that dispersibility of this product could be increased further by reducing the oversize fraction;

7. Since the weight fraction and dispersibility of each size fraction is known (Figures 24 and 25) 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 works out at 52.7%. In comparison, the measured dispersibility of a sample of this granulation is 52.4%. This close agreement emphasises the importance of particle size in influencing dispersibility;

8. Figure 24 serves well to illustrate the importance of both particle size and rewetting moisture upon the dispersibility of the final granulated product. This is shown by the fact that the 14% rewet granulation exhibits a lower dispersibility than the 11.6% rewet product at any particular particle size. For example, at 400/u size the former product has a dispersibility of 25.3% while the 11.6% rewet granulation has a dispersibility of 67%.

(iii) Uniformity of Moisture Distribution and Effect of Mixing During Granulation

It has already been shown how the technique of wet granulation may be applied to NFDM in order to simulate an instantising or agglomeration process. Using this technique optimum conditions for reconstitution have been determined with regard to rewetting moisture and particle size.

A question which arises, however, when comparing the dispersibilities of different size fractions is the possibility that the lower dispersibility of large size fractions (e.g. Figure 24) may be due to that fraction having possessed a higher moisture content at rewetting. This could arise due to non-uniform moisture distribution

during mixing and granulation. To some extent it appears unlikely that gross non-uniformity in moisture distribution is occurring since such a sharp and reproducible Break Point is obtained with this wet granulation technique. However, as was seen from the literature review considerable attention is paid to the method of moisture addition in commercial processes. It is reasonable, therefore, to study the efficiency of moisture addition in this simulated technique. Since the time of mixing may also have an effect upon this question of moisture distribution it was decided to examine this point as well.

Three batches of Niro NFDM were wet granulated and treated as follows :

(a) One batch was rewetted to 11.8% moisture corresponding to Break Point rewetting. It was granulated employing the normal procedure as far as the formation of moist "granules". These were not dried but were sieved in the moist state to yield three fractions, which were tested for moisture content;

(b) A second batch was rewetted to 11.8% moisture as in (a) above but in this instance an additional 30 minutes mixing was given after all the water had been added to the NFDM. The mix was then granulated and sieved in the moist state as for (a);

(c) The third batch was rewetted to 13.7% moisture which is well above Break Point rewetting. The normal procedure was employed and three fractions were obtained from sieving of the moist granules.

The results for these three batches are presented in Table 8.

TABLE 8

Sieving of Wet Granulated NFDM in Moist State

Description of Sample	Weight Fraction	Moisture	Nature of Fraction
(a) 11.8% Rewetting Normal Procedure			
+ 850/u Fraction	21%	11.5%	Granules
640/u Fraction	74 *	11.6 *	Fluffy powder*
- 420/u Fraction	5 *	11.6 *	Fluffy powder*
(b) 11.8% Rewetting Additional 30 mins. mixing			
+ 850/u Fraction	19%	11.9%	Granules
640/u Fraction	21	12.0	Granules
- 420/u Fraction	60 *	12.1 *	Fluffy powder*
(c) 13.7% Rewetting Normal Procedure			
+ 850/u Fraction	52%	13.5%	Granules
640/u Fraction	32	13.7	Granules
- 420/u Fraction	16	13.4	Granules

* Fluffy powder in these fractions cannot be sieved satisfactorily since blinding of screens occurs.

The following points may be enumerated from the results :

1. It is seen that no size fraction has been separated having any marked difference in moisture content. The largest difference between fractions noted is 0.3% between the 640/u fraction and the less than 420/u fraction in (c).

This certainly suggests that mixing is sufficient to promote uniform moisture distribution on rewetting;

2. As would be expected sieve separation is most difficult in a moistened powder, while it is still in the "fluffy" state. This leads to blinding of the screens as for example in the 640/u fraction of (a) where only 5% was able to pass the 420/u screen. Where the fraction separated consisted of fluffy powder this has been indicated by an asterisk. As soon as a granular product is formed, however, excellent sieve separation is possible with a minimum of blinding, e.g. as in (c);

3. Since 11.8% rewetting corresponds to Break Point conditions it is to be expected that some granules are formed although as seen in (a) and (b) the majority separated consists of fluffy powder. Nevertheless, the granules separated did not possess a higher moisture content;

4. Upon additional mixing for 30 minutes as in (b) it is seen that a granular fraction was separated above the 420/u screen whereas this did not occur with (a). This suggests that there is present a higher portion of granular product in (b). However, it may also be noted that all fractions in (b) possess a slightly higher moisture content, presumably due to equilibration with the atmosphere during the additional mixing period. This means that the additional granule formation noted in (b) may be due either to the slight

moisture uptake or to the additional mixing performed. Certainly the possibility exists that the degree of mixing may influence granule formation particularly in the Break Point region of rewetting.

(iv) Additives in Wet Granulation

It was mentioned in the literature review that one team of workers, Moore et al (1964) have recognised the potential applications of the use of additives in commercial instantising. One of the main problems of research into this field is the difficulty of establishing controlled trials with commercial scale equipment. However, use of a simulated process such as this technique of wet granulation makes possible a study of the effect of additives on product characteristics such as dispersibility. Obviously, the area of additives is an immense field and only an introduction is intended here so as to demonstrate how the technique may be used in such research.

Batches of Niro NFDM were wet granulated as described in PROCEDURE, using the 1600/u screen. Four additive treatments were examined, each being compared with a control curve for granulation with water only. Results are presented in Figures 26,27,28 and 29.

(a) Rewetting with 10% Lactose Solution

Lactose is commonly employed as a binder in the pharmaceutical industry. However, in the case of NFDM approximately 50% of the powder composition is lactose so it would be expected that sufficient binder of this type is already present. But it is not clear what relationship exists between the lactose and the water of rewetting during

granulation since at least two distinct possibilities exist :

1. The Break Point in rewetting may occur at a point where sufficient binder solution has been formed, by the solution of lactose, to allow adhesion of particles. If the formation of this binder solution were a limiting factor in granulation then it would be expected that a Break Point would be obtained at lower moistures, where rewetting with lactose solution;
2. The Break Point in rewetting may depend largely upon the volume of liquid added rather than on its binding properties, particularly if formation of a binder solution is not a limiting factor. If this is the case then little shift of the Break Point would be expected on rewetting with lactose solution.

From Figure 26 it is seen that, in fact, rewetting with 10% lactose solution has resulted in little significant change in the Break Point, or in the nature of the dispersibility curve. This would suggest that the availability of lactose to form a binding solution during granulation is not a limiting factor.

(b) Addition of 5% Lactose Powder to NFDM

In this case 5% commercial lactose powder was added to the NFDM prior to granulation using water for rewetting in the normal manner.

It is seen from Figure 27 that this treatment has resulted in a slight shift in the Break Point and a modification of the dispersibility

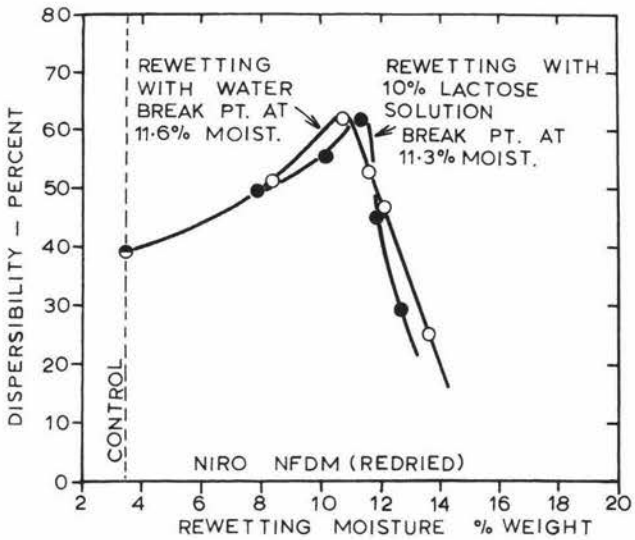


FIG. 26 EFFECT OF ADDITIVES — 10% LACTOSE SOLUTION

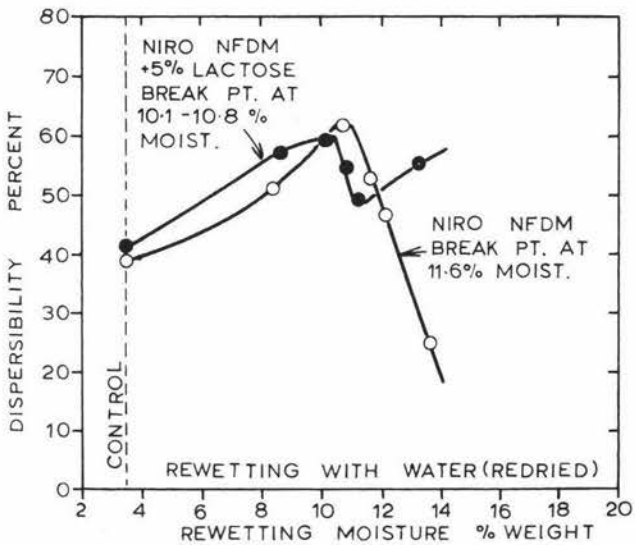


FIG. 27 EFFECT OF ADDITIVES — 5% LACTOSE TO NFDM

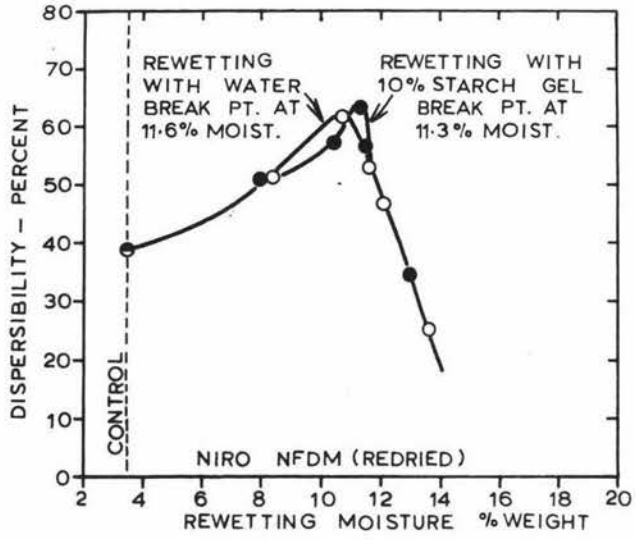


FIG.28 EFFECT OF ADDITIVES — 10% STARCH GEL

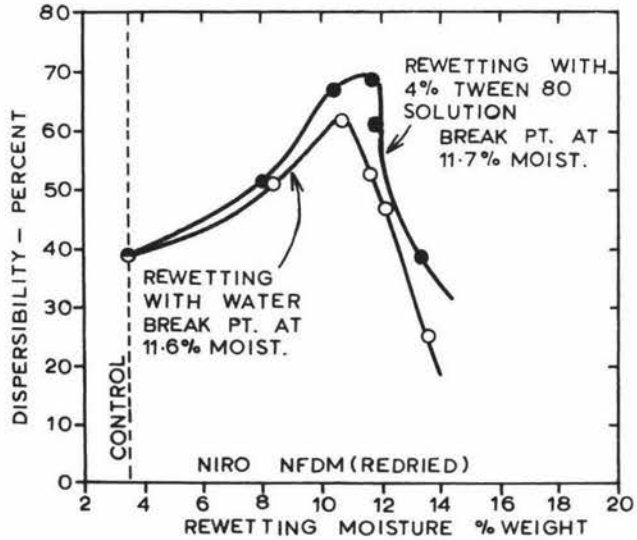


FIG.29 EFFECT OF ADDITIVES — 4% TWEEN 80 SOLUTION

nature of the curves in both cases are very similar. This again would suggest that sufficient binding substances are present in NFDM in order to achieve granulation.

A point of interest here is the fact that wet granulation was achieved in this case by the addition of a GEL, i.e. the water was held within a gel network. Yet even so, the same quantity of water was required, there being no shift in the Break Point for granulation. This emphasises the critical nature of the water in the mechanism of granulation.

(d) Rewetting with 4% Tween 80 Solution

As discussed in the literature review other workers have investigated the effect of surfactant addition to milk before drying upon the dispersibility of the milk powder. In this study, surfactant was added per media of the rewetting liquid during granulation. The surfactant chosen was Tween 80 which represents a rating of 15.0 on the Atlas H.L.B. System. (Atlas Chemical Industries Inc. (1963)). The H.L.B. system rates surfactants from a value of 0.0 for the most lipophilic to a value of 20.0 for the most hydrophilic. Use of 4% Tween 80 represents approximately 0.4% Tween 80 by weight of NFDM solids at Break Point rewetting. This compares with levels employed in coffee whiteners (Atlas Chemical Industries Inc. (1965)).

From Figure 29 it is seen that addition of Tween 80 results in a significant improvement in the dispersibility of the granules formed. The Break Point for granulation is not shifted. It may be predicted that this improvement in dispersibility is brought about by

a reduction in surface and interfacial tensions tending to inhibit rapid dispersion in the control. The fact that an improvement in dispersibility extends also to the above Break Point rewetting granules suggests that surfactant may actually assist penetration of water into the granules, i.e. in the nature of a dispersant.

It must be emphasised that Tween 80 is only one of an immense range of surfactants. Although it has been shown that dispersibility can be improved with this additive it may well be that other surfactants, or blends of surfactants, may be even more suitable for this application.

E. PRINCIPAL RESEARCH FINDINGS

1. A granulation procedure is described which has been successfully applied to NFDM. As this technique, is in effect, a simulated instantising process it may be employed to study the effect of certain variables upon the properties of granulated NFDM;
2. It is shown that the two major factors influencing the reconstitution properties of granulated NFDM are the rewetting moisture content of the powder before redrying and the particle size of the granules;
3. The rewetting moisture content is shown to be a most critical variable, the optimum being approximately 11-12% moisture for the NFDMs studied. This optimum corresponds with maximum reconstitution properties as well as the Break Point in granulation;
4. The effect of particle or granule size upon reconstitution characteristics of NFDM has been clearly demonstrated. Optimum size has been found to be approximately 200/u; coinciding with optimum dispersibility and infinite sinkability;
5. The rewetting technique employed appears to be satisfactory since a uniform moisture distribution has been demonstrated in different size fractions of a rewet granulated NFDM;
6. Examples have been given of the manner in which this granulation procedure may be employed to study the effect of additives in attempts to improve the dispersibility of

powders. A significant improvement in dispersibility can be achieved by rewetting with Tween 80 solution on instantising as compared with rewetting with water alone.

IV. FIELDS FOR FURTHER RESEARCH

1. Azeotropic distillation of certain food powders has been shown to consist of two phases, viz.: (1) a rapid initial moisture desorption and (2) a slower desorption corresponding to a first order reaction. In the case of NFDMs this first order reaction has been attributed to the dehydration of crystalline lactose hydrate as suggested by Choi et al (1948) while in the case of tomato powder this may be due to a caramelisation or browning reaction to produce water. Further research on the nature of this first order reaction appears warranted since the change in desorption from phase (1) to phase (2) may provide a division mark between desorption of "free water" and "bound water" respectively, thus providing a relatively easy determination of free water in a powder;
2. It has been shown that lactose may exist in a crystalline state in commercial NFDM under present day manufacturing conditions. Research is suggested to assess technological implications of lactose in a crystalline state as compared to the glassy state;
3. A working theory has been proposed to explain the mechanism of dispersion of a soluble food powder. Further applied research may enable modification or enlargement of this theory, while rather more fundamental research would enable quantitative theory to be developed for certain aspects of dispersion. For example, the influence of particle size

upon sinkability of a powder has been clearly demonstrated in this research. It has been shown that a critical particle size exists for a powder above which the sinkability index tends to infinity. This observation suggests that it should be possible to construct a quantitative model to determine critical particle size in terms of particle density, surface tension, interfacial tension, etc. However, a lack of fundamental data of interfacial tensions in these powder/water systems has made this impossible;

4. No significant increase in dispersibility of NFDM has been attained following a compression/regrinding process. However, it is felt that if a suitable non- aqueous adhesive could be found then such a compression technique may well be employed to promote aggregation of particles and thus increase dispersibility. For example, propylene glycol is employed in the tabletting of dry ingredients to form a popular dog "biscuit" in U.S.A. Research with this type of liquid adhesive may be fruitful;

5. It has been shown that the bulk density of a spray dried product such as instant coffee can be markedly increased by a compression treatment without any adverse effect on reconstitution characteristics. Research is warranted with other spray dried products which show similar "balloon" particle structure to ascertain if the technique can be extended. In particular the effect of compression upon spray dried sodium caseinate deserves attention;

6. Results obtained on compression of instant coffee indicate that an improvement in the dispersibility of the powder has been achieved through modification of particle structure. This suggests the desirability of research to define optimum spray drying conditions corresponding with optimum reconstitution characteristics; particularly in view of the fact that some saving in packaging volume appears possible;

7. The optimum temperature of water for reconstitution of NFDM has been determined to be approximately 50°C. A similar procedure may well be employed to study optimum reconstitution temperatures for other food powders;

8. It has been noted that the addition of free flow agents to NFDM appears to involve a compromise between improved flow properties and a slight decrease in dispersibility. However, research appears warranted with a wider range of such flow conditioning agents. Further, an objective measure of flow properties of a powder would be desirable in such a study, as for example, angle of repose;

9. A technique of wet granulation has been developed which simulates the instantising or agglomeration process as applied to NFDM. Although this technique has only been applied to NFDM in the present study a wide field of research remains open in the application of granulation to other foods powders as a means of improving dispersibility and flow characteristics;

10. The critical nature of rewetting moisture as a variable in the granulation, or agglomeration process has been clearly demonstrated. More fundamental research may elucidate the role of water in granulation of NFDM. In particular, it would be interesting to clarify the reasons for increased insolubilisation of NFDM when granulated at above Break Point moistures;

11. It would be expected that in commercial instantising of food powders a major portion of the manufacturing cost would be represented by the redrying operation. This suggests the desirability of reducing the optimum rewetting moisture content of the process. Research may be profitably directed towards a means of reducing the amount of water to be added in granulation; such as by use of binders/adhesives or use of non-aqueous solvents;

12. Only a very introductory examination of the field of additives in granulation has been made in the present study. Even so, the benefits to be gained by the use of a surfactant such as Tween 80 has been demonstrated. Further research with other additives and surfactants and determination of optimum concentrations of such additives may well yield even greater improvement in the reconstitution characteristics of powders and granules.

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