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.

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July, 1967.

"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

ACKNOWLEDGEMENTS

I wish to express my sincere thanks to my supervisor, Mr H.A.L. Morris, for his advice and guidance, and the stimulation of enthusiasm which he provided by his constant willingness to discuss aspects of the research.

I also wish to gratefully acknowledge :

* Mr G.B. Latimer for much valuable advice, discussion and criticism;
* Dr. R.M. Dolby, of the New Zealand Dairy Research Institute, for his continual interest and for providing the opportunity for much valuable discussion;
* The Australian Dairy Produce Board for the award of a Studentship which made this study possible;
* The Department of Agriculture, Victoria, for the granting of leave of absence such as to make this study possible;
* Miss S. Cooper for her patience and skill in the typing of this thesis;
* Miss D. Scott, of the Central Photographic Unit, for much of the photography and all of the reproduction of figures in this thesis;
* Mr G. Burns, of the Faculty of Veterinary Science Photographic Unit, for the processing of proofs of the microphotography undertaken;

* The New Zealand Dairy Research Institute for valuable discussion with various staff members and for making available powder sieving facilities.

Finally, I wish to record my gratitude to my wife, Felice, for her constant help and encouragement throughout this work.

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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

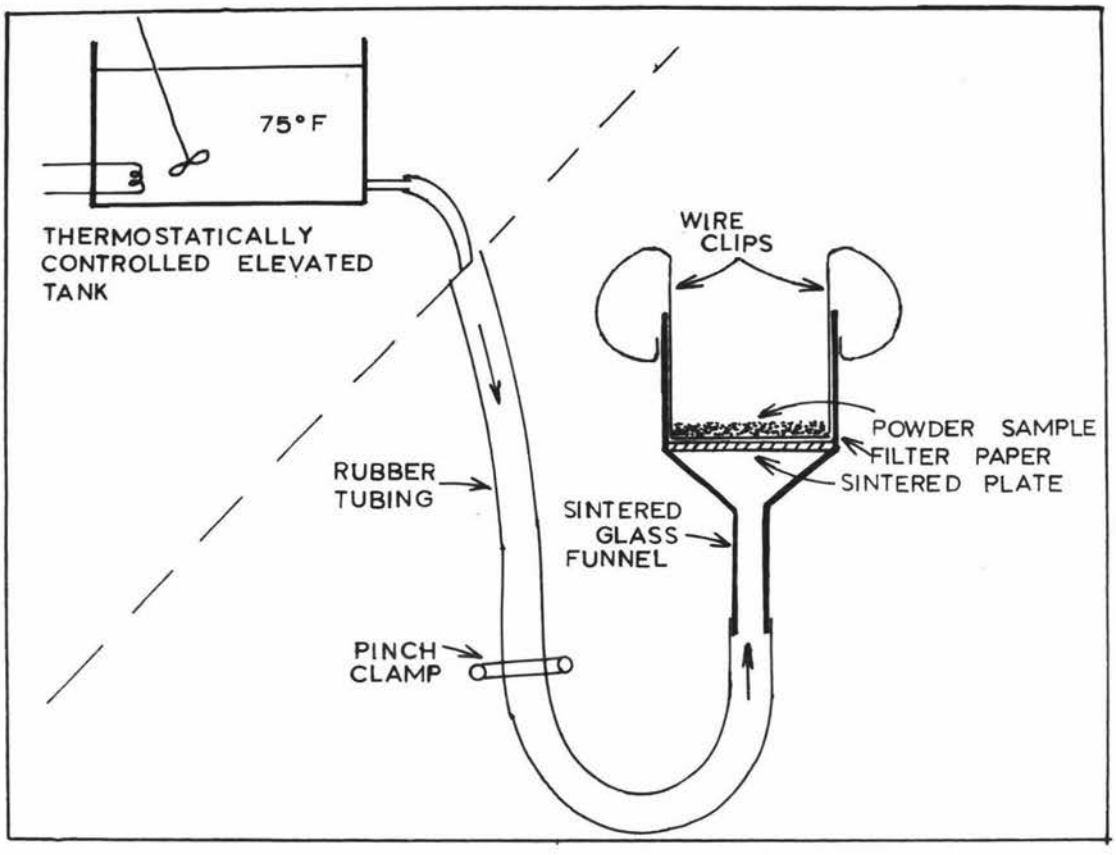

FIG.1 APPARATUS FOR SINKABILITY INDEX DETERMINATION

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

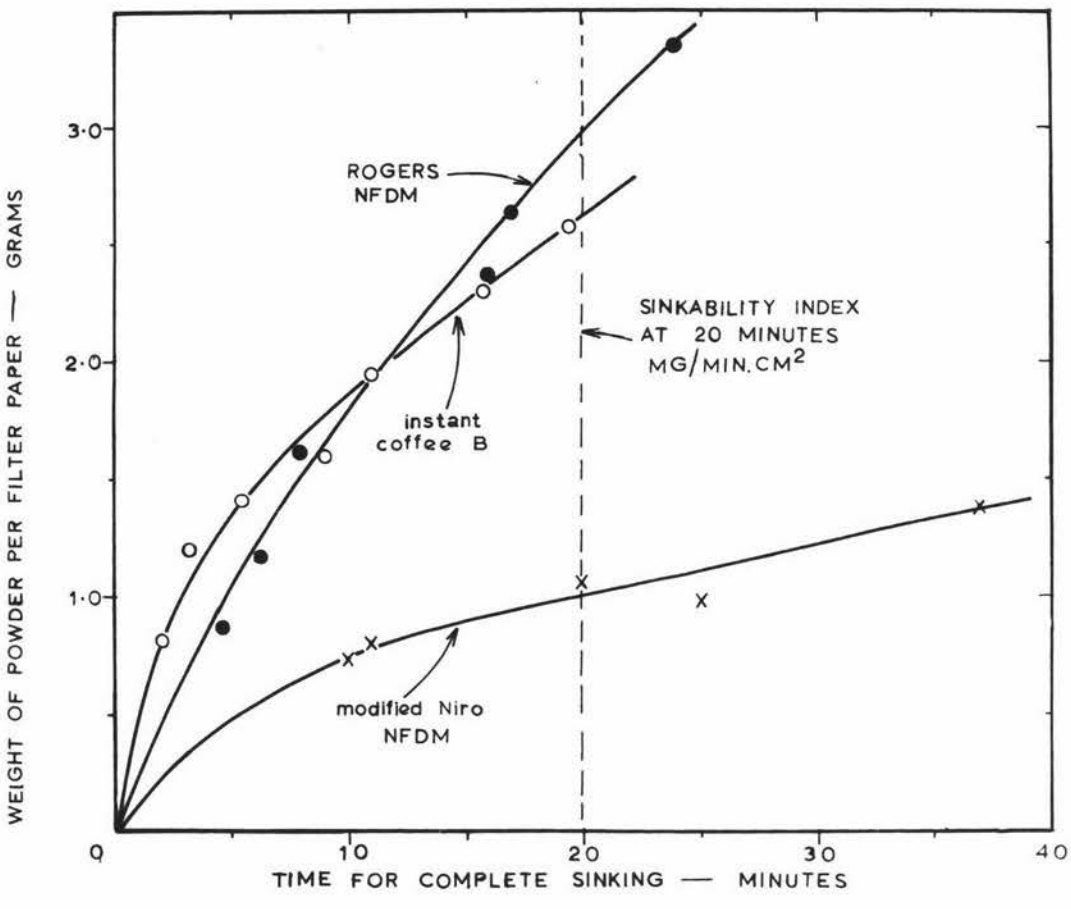

FIG. 2 TYPICAL SINKABILITY CURVES

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

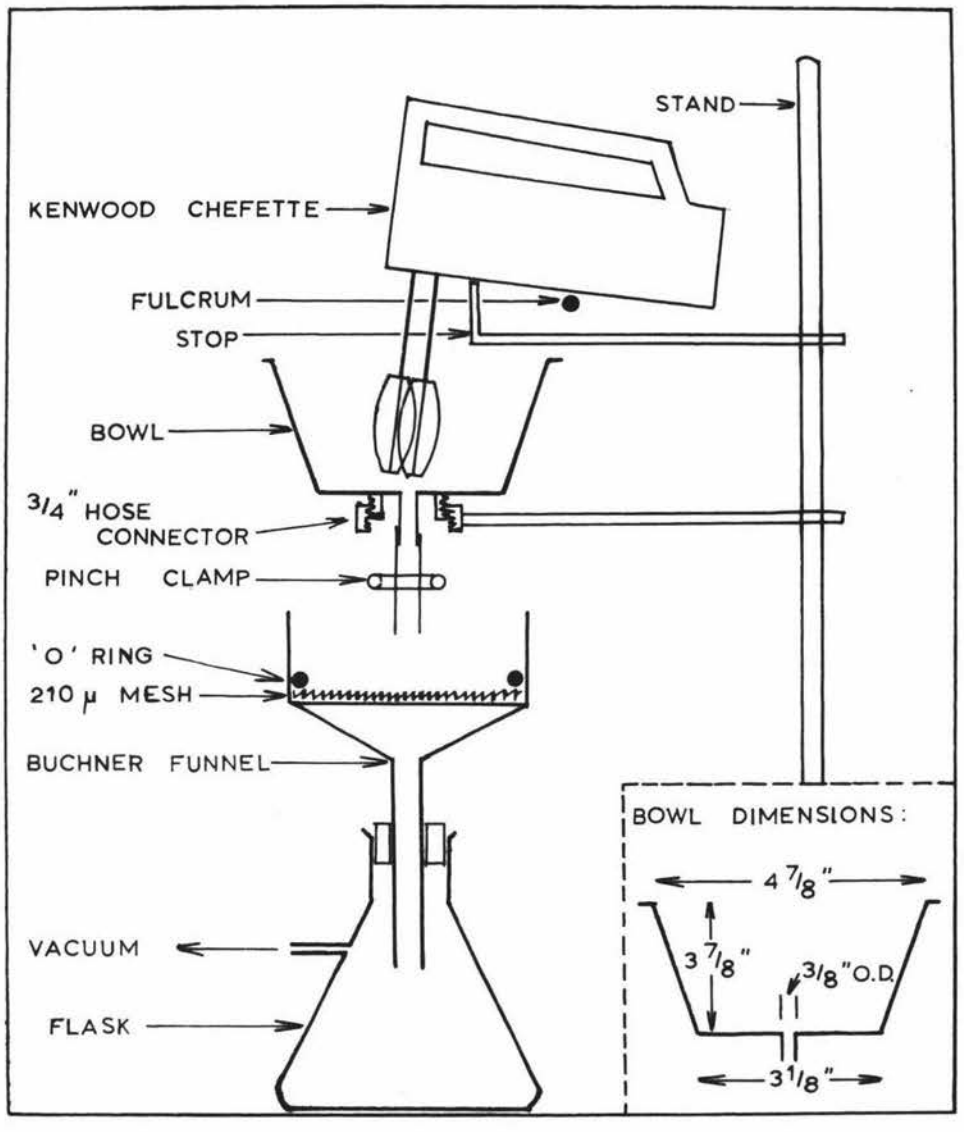

FIG. 3 APPARATUS FOR DISPERSIBILITY TEST

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

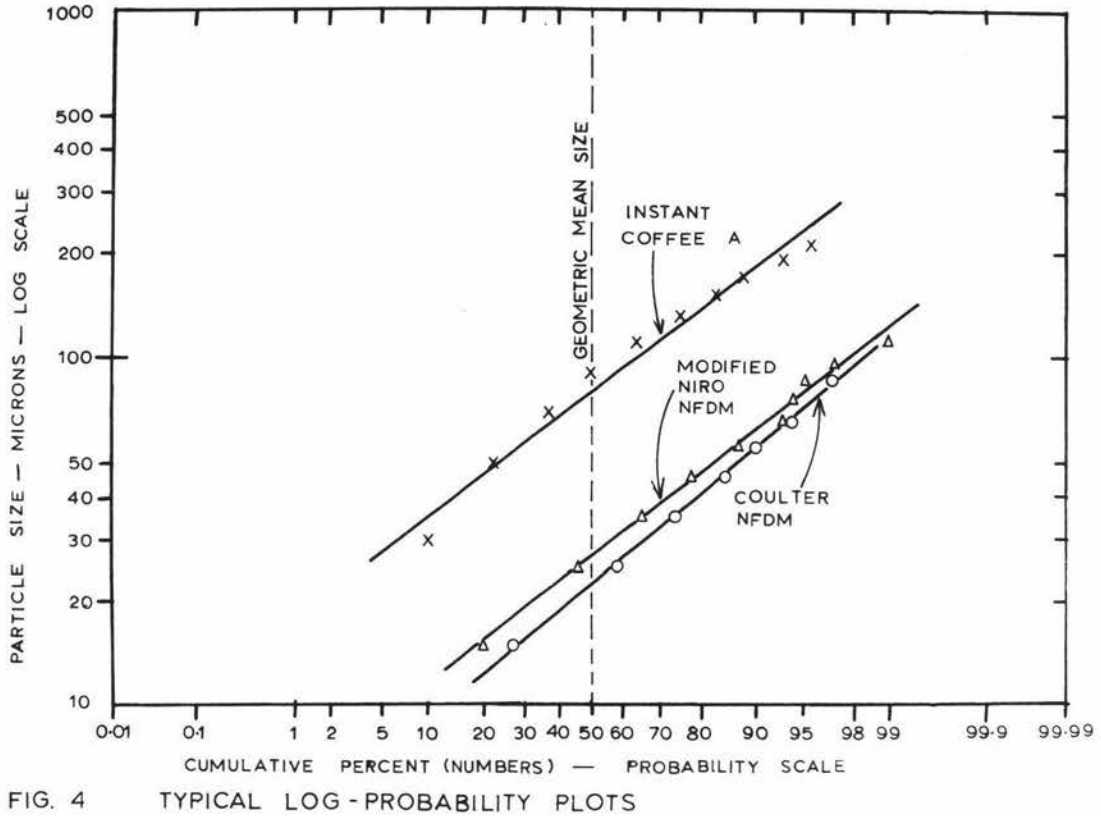

FIG. 4 TYPICAL LOG-PROBABILITY PLOTS FOR PARTICLE SIZE ANALYSIS

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.