STUDIES ON PHYSICAL CHARACTERISTICS OF ROLLER/SPRAY DRIED, INSTANTISED BUFFALO'S MILK POWDER AS INFLUENCED BY CONDITIONS OF MANUFACTURE AND STORAGE

A Thesis
Submitted to the Mahatma Phule Agricultural University for the Degree of
DOCTOR OF PHILOSOPHY

By
UTTAM INGLE
M.Sc. (Agri.) Dairy Science
Division of Dairy Technology
NATIONAL DAIRY RESEARCH INSTITUTE
Karnal (Haryana)
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I certify that the work reported in the thesis entitled 'Studies on Physical Characteristics of Roller/Spray Dried, Instantised Buffalo's Milk Powder as Influenced by Conditions of Manufacture and Storage' was carried out by Shri Uttam Madhvrao Ingle, M.Sc (Agri.) Dairy Science, for the requirement of the Degree of DOCTOR OF PHILOSOPHY in the Faculty of Agriculture, Mahatma Phule Agricultural University, Rahuri, under my guidance.

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Introduction
INTRODUCTION

Till the year 1951 almost our entire requirement of dried milk and related products were imported. Development of the Dairy Industry on organized lines commenced with the successive five years plans. Since the beginning of Second plan, 8 factories for milk powder have been established mostly in private and co-operative sectors (Souvenir 6th Dairy Industry Conference, Chandigarh). Recently 5700 tons of milk is being manufactured for domestic consumption. Although in early stages, the development of dried milk industry was based on imported know-how, the time has come when sustenance and further development of the industry will depend on locally developed knowledge on the subject. It is worth considering in this connection that almost exclusively buffalo’s milk is employed for the manufacture of dried milks.

It is now well understood that the characteristics of buffalo’s milk vary considerably from those of cow’s milk. As a result the quality of the dried products from the buffalo’s milk is bound to differ from similar products made out of cow’s milk.

We have no knowledge of micro-structure of individual particles of buffalo’s milk powder which
has been manufactured by various processes. The
distribution conditions of the casein particles, fat
globules, lactose and air in individual particles of
the powder play an important part in determining the
various physical attributes. Among such attributes
special reference may be made to particle size, bulk
density, solubility, wettability, sinkability, dispersi-
bility and colour. These characteristics are major
points for consumer's appeal and depend both on the
quality of basic raw material as well as the method of
manufacture and condition of storage. It was, therefore,
considered desirable to undertake the study mainly
concerned with the above mentioned physical attributes
of buffalo's milk powder prepared by roller, spray and
instantisation processes.

Conventionally made skim milk powder has never
been well received by housewives for beverage purposes
to the difficulty with which it constitutes in
cold water, its poor penetrating property in water
and the tendency to cake and dust. These drawbacks
of regular powder led to the advent of 'Instant' milk
powder which has revolutionised the dried milk industry
since its introduction in 1954. The ready dispersibility
of dried milk which was earlier sought to be achieved
in many research laboratories by rewetting process found
expensive. Peebles (1964), who is inventor of instantizing process.

Since 1954, the production of 'instant' non-fat dry milk has increased rapidly. Practically all non-fat dry milk for home use in U.S.A. is now instantized. This came to 253.3 million lbs. or 28.5 per cent of the total non-governmental use in 1963 (Mori and Hedrick, 1965). The phenomenal development offers new avenues to the utilisation of dried skim milk produced in the world that has increased from 1,324,000 tons in 1964 to 1,972,000 tons in 1968 (Charles, 1969) of which approximately 53 per cent is produced in America and 33 per cent is produced in North-West Europe. The largest of the world's exporters are the U.S.A., France, Denmark and Holland. India has, particularly, for many years imported substantial amounts of skim milk powder. In India alone the import of dried skim milk was 31,759,27 tons in 1967-1968 (Souvenir, 6th Dairy Industry Conference, Chandigarh), it is envisaged to be 126,000 tons during the next five year under the World Food programme to meet the country's need (Robinson, 1969), which excludes milk powder to be imported from sources such as CAG, FAO, WFP, etc. for special aid programmes.

In spite of large production, meagre research has been published on the processing factors which
influence the characteristics of final instantized product and the methods of control. There is no doubt some commercial experience has provided some information, but dissemination of it has been meagre.

Modern organised dairying in this country is depending almost wholly on milk from buffaloes which constitute about 52 per cent (F.A.O., 1968) of the total milk produced in the different states of India. The higher yield together with its richer fat contents has led to the preferential utilisation of buffalo's milk for products.

Most of the manufacturing methods for conventional dairy products like milk powder have been developed in countries with moderate to cold climate for cow's milk only. Modern dairy equipments are designed and fabricated to work these processes. Adoption of imported know-how for handling buffalo's milk with foreign designed equipments has, therefore, brought in its wake problems of product quality and out-turns. The roller dried skim milk powder manufactured from buffalo's milk is poorer in yield as well as reconstituability as compared to that produced from cow's milk due to high initial viscosity of the concentrated milk (Sat Prakash, 1965). Moreover, the economic utilization of seasonal buffalo's milk is a problem of significance to the dairy industry for its viability and demands for
conversion of such surplus buffalo's milk into easy-to-
reconstitute powder to even out the regional imbalance.

With the growing demand for milk in the country, combined
with the limited scope for immediate improvement of
supplies, it is expected that more and more reliance
will be placed on the tonning of buffalo's milk with
skim milk powder made during flush season and for this
purpose the skim milk powder of high dispersibility and
quick 'fall-in' time is a prerequisite. The present
investigation has, therefore, been undertaken to
standardise the method for the production of instant-
non-fat buffalo's milk powder using Anhydro spray drier
fitted with accessories for 'single-pass' instant powder
production.

The properties of 'instant' powder and methods
of its manufacture are still very much discussed and there
seem to be considerable differences of opinion as to which
type of 'instant' milk powder is the best and the prefer-
able method of its manufacture. The term 'INSTANT' has
been and still is very much misused due to lack of proper
definition of instant powder and lack of official
standards for the products and a method to measure them.
The International Dairy Federation has been appointed
a working party under the Chairmanship of Dr. Crossley
to examine the methods for measuring wettability,
dispersibility and solubility of 'instant' skimmed milk powder. The position today is that 'instant' skimmed milk powder of satisfactory quality is being manufactured on commercial scale, the manufacture of 'instant' whole milk powder is still 'around the corner'.
Review of Literature
Review of Literature
1. History of Dried Milk

Dry milk has been known in some parts of the world for many centuries. Marco Polo in the 13th century had reported that soldiers of Kublai Khan carried dried milk on excursions. Before it was used, water was added to a portion of the dried material. Mixing was accomplished by the movement of the horses during riding on the trip. The recombined product was then consumed at meal time. The belief is that part of the fat was removed from the milk before drying and dehydration was accomplished by solar heating (Eckles et al., 1961).†

† The Japanese had a concentrated milk product in the 7th century but there is no recorded reference to this dry product (Miyawaki, 1928). In the United States, the development of evaporated or concentrated milk and dry milk as known today began in the early 1800s. But progress was slow during that century. In 1809, Nicholas Appert, a Frenchman, announced his discovery that milk could be reduced to one-third the original volume evaporation of the moisture from an open vessel. Cooking followed by sealing the product in a container greatly enhanced the keeping quality. He also developed a dried milk in tablet form by air
drying of milk solids concentrated to the consistency of a dough (Olson, 1950).

Gail Borden was one of the leading pioneers in originating a process of milk condensing (Goodeale, 1956). By applying a partial vacuum to remove moisture from milk, he obtained a product that was superior in flavour to the one obtained by boiling in open vessels. Although initially, he encountered difficulties in marketing condensed milk, persistence eventually resulted in a commercially successful business.

Meyenberg (1884,1887) who came to the United States from Switzerland, introduced a unique concept for improving the keeping quality of evaporated milk. He obtained patents in 1884 and 1887 for sterilizing evaporated milk by heating to 240°F, under pressure with steam while the sealed cans were in continuous motion. This innovation provided the basis for a new industry.

During the last half of the 19th century attempts to produce a dried milk involved the addition of other dry products to concentrated milk. A British patent was granted in 1855 to Grimwade who developed a modified dry product from highly concentrated milk to which was added sodium or potassium carbonate and sugar (Hunziker, 1949). This semi-solid material was extruded into thin streams and dried in trays. Malted milk powder was made from whole milk, extract from malted
barley and wheat flour. Others who pioneered in methods of moisture removal from milk were Reine, Newton, Horsford, Daison, Gallois, and Dacuwe (Miyawaki, 1928).

Patents and reports which emphasized processes for dry milk manufacture without the addition of other products began about 1898. In 1901, Campbell of the United States and Hunsiker of Denmark dried concentrated milk on trays (Hunsiker, 1949). Later they used concentrated milk and a steam heated cabinet. In 1902, Hall obtained a patent on a new system of manufacturing dry condensed milk.

Just (1902) was among the first inventors to receive patent rights on a drum drier with two rolls. Hatmakers of England improved Just's model. These types included Gathmann's single cone drum with a grooved surface and Mignon Plume's drum with a smaller unheated drum for film application to the larger drum (Hunsiker, 1949). Vacuum drum dryers were designed by Ekenberg, Sweden (1889), Passbary, Germany (1903) and Grovers (1909).

Among the early inventors of spray drying equipment was Perry, who in 1872 combined atomization of a fluid and heated air. Stauf, a German, received a U.S. Patent on an improved design based on this principle in 1901. Four years later Macalachen (1905) processed dried products by atomizing milk, skim milk, eggs or
blood in heated air. Improvements and modifications of spray drying technique were developed and patented by Marrel et al. (1907); Gray and Jensen (1913); Rogers (1917) and Beardslee (1948). Gray and Rogers are outstanding contributors towards the technical development and commercial application of the spray drying process. Another was Roger who did much for the industry's growth with his engineering improvements and fabrication of spray drying equipment.

The advent of the instant milk powders has probably been the most exciting event in the history of the North American dried milk industry since the introduction of spray drier. While this type of milk powder had not been marketed widely in Europe as yet, it has caused a revolution in the dried milk industry of the United States and Canada.

Instantizing was designed to improve the dispersibility of spray dried milks. Since the introduction of instant skim milk powder in 1954, the sale of regular skim milk powder in the retail market has been completely replaced by the instant products. Regular skim milk powder was never well accepted by the housewife for beverage purposes due to the difficulty with which it reconstituted, its poor pouring characteristics and its tendency to cake. These drawbacks were overcome by instantizing the powder.
Peebles, of the Western Condensing Company of California, must be considered the pioneer in the development of instant milk powder. His process (Heivighorst, 1954, Peebles, 1956 a,b and 1958) was first introduced commercially in 1954. The product is often referred to as a two-stage instant as its manufacture consists of the controlled moistening and redrying of regular spray-dried skim milk powder. Single stage instant refers generally to instant powders dried from liquid milk in a single drying operation. When used for instantizing, the regular spray-dried product is often referred to as the base powder.

Peebles' method consists of delivering base powder to the top of a vertical agglomerating chamber. In this chamber the particles come into intimate contact with finely atomized water and air saturated with steam. The moistened particles stick to each other to form aggregates as they fall through the chamber into a conveyor belt. The moist powder containing 10 to 20% moisture is then conveyed to a shaker-type drier where the moisture is reduced to the desired level of 4.5-4.0%. During the process a substantial part of the lactose changes to a crystalline form.

Lauder and Hodson (1958) likewise were granted a patent for the manufacture of an instant milk powder.
Their method provided for instant wettings of base powder with steam to a moisture content not exceeding 9.0% and preferably 5.5%. This is achieved by causing a flow of steam to strike a falling stream of powder transversely. The resulting agglomerating moist powder immediately contacts a stream of hot air, which, according to claims, instantly dries the agglomerated powder and carries it horizontally from the chamber. It is said by inventor that this instantized product is characterized by a relatively high \( \alpha \)-lactose content and a low \( \beta \)-lactose content. This is thought to improve the dispersibility of the powder.

Patents were granted to Biswell (1959, 1960 a,b) for a steam instantizing process. Base powder is fed into a moving belt from which it falls as a curtain into the path of a row of steam jets. The steam wets the powder and simultaneously projects it into a chamber through which a second belt passes. The aggregates fall on the belt and are carried into an adjacent drying chamber. It is claimed that a substantial portion of the lactose in the base powder, which is in the \( \alpha \)-anhydride form, is changed to the \( \beta \)-hydrate form in the instantized product. In the Cherry-Surrell system (Carlson et al. 1953) base powder is agglomerated in a horizontal chamber with moist air to a moisture
content of 6-10%. The wetted particles pass to a cyclone collector from which they fall into a moving stream of air heated to 270°-300°F. The dried aggregates are sized between steel rollers and then packaged. Griffin (1959) was granted a patent for an instantizer which is cone-shaped and very compact. Base powder is caused to fall as a curtain between two steam jets. The powder is instantly wetted and falls into the top of the instantizer where it encounters heated turbulent air. The turbulent air causes the powder particles to agglomerate into clusters and also removes the moisture previously picked up from the steam jets. The instantized product falls into a moving belt which carries it away for cooling and packaging. A French patent was also granted for this process (Blaw-Knox Co. 1957). Scott (1959) received a patent for a novel method of agglomerating wetted milk powder. Base powder is wetted in a hydration chamber with a fine spray of water or milk to a moisture content of 10-14%. It is then conveyed to a hammer mill during which time (15-20 sec) the wetted protein is said to swell and the milk sugar to hydrate and absorb its water of crystallization requirements. The purpose of the hammer mill is to break up the large wetted chunks of powder which may form in the conveyor coming from the hydration vessel. Also, the impact of the blades
in the mill is said to cause the agglomeration of the particles into clusters or granules of the desired size. The powder is then redried on porous belts which travel through drying ovens at temperature between 160° and 240° F. †

It is mostly skim milk powder that is being instantized at the present time. However, it has been reported by Hall and Hedrick (1961) that small amount of instantized whole milk powder is being marketed in the U.S.A. as well as a partially skimmed milk powder containing 17% fat is being marketed in Canada. Keeping quality of the fat-containing products is the chief deterrent to increased production as the fat is prone to oxidative changes unless special precautions are taken.

A patent which provides specifically for the manufacture of an instant fat-containing milk product was granted to Peebles (1959). His method requires several steps. Skim milk was first spray-dried and then instantized as already described (Peebles, 1959). Fat was then added by allowing warm powder to fall through a chamber in which the melted fat was absorbed into the porous structure of the agglomerated skim milk powder. It is claimed that the product is readily dispersible in water.

Sjollema (1960) described a method of coating
skim-milk as whole milk powder with a surface active agent such as oleic acid or soya-lecithin to produce a product with instant properties. Warm water is mixed with the liquid surface active agent in an amount of 2% by weight for about 5 minutes and is then cooled. It is claimed that the finished product shows excellent instant action in cold water even after a year of storage provided the lactose is not allowed to crystallize and thereby de-emulsify the milk fat.

The instantizing process discussed thus far have required that a base powder be reprocessed. This reprocessing of the powder increases the cost of the final product and in some instances may have a deleterious effect on flavour and solubility. It was thought that these objections would be overcome by producing an instant product in a single stage (or by a single pass) without the need for reprocessing base powder. Considerable success has been achieved in producing the so-called single stage 'or single pass' instants which include both the large particle instant and the single stage agglomerated instant.

Specially constructed or modified spray-driers are usually necessary to produce single-stage spray dried instants. It is obvious that in order to dry a larger particle, longer drying times or higher drying
temperatures are required. Since the heat-damage of protein places a limit on the higher temperatures that can be used, a longer drying time is often employed. A tower type drier which allows the particles to remain suspended in the drying air for longer periods of time is ideal for this purpose.

Coulter and Townley (1959) designed a drier which can be operated to produce a large particle instant powder by varying conditions of drying such as nozzle size, feed pressure, and drying temperature.

Emundson (1961) described and designed a tower type of drier as tower was suitable for the production of large particle instant or single-stage agglomerates.

Sharp and Kempf (1960) patented a method of manufacturing instant milk powder without having to re-wet the base powder. Several steps are required to produce the desired agglomeration of the dried milk. Milk concentrate that has been seeded with lactose crystals is sprayed into a drying air at a temperature of 240-250°F. The powder is removed from the chamber to cyclones as the moisture content reaches 18% or slightly less. The powder falls to a scroll conveyor which delivers it to an air convector at moisture level of 12-13%. Air, heated to 185-200°F carries the powder to a cyclone separator from which it is
discharged at 8-9% moisture. The powder is then delivered to a tray-drier which reduces the moisture to 2-3%.

Sanna (1960) invented a tower type spray drier which is said to be suitable for producing single stage instant milk. Atomize (1962) invented an instantizer. This agglomerating unit, suitable for the production of instant milk, is coupled directly to the outlet of a spray drier. It is in the form of a 3-section funnel accommodating a 3-section vibrating conveyor extending its full length. The product containing at least 8% moisture, is agglomerated in the first section by warm air the vibration preventing its fusion; simultaneously as a result of the crystallization of some constituent, the particles lose the tendency to stick together, some moisture also evaporate but the drying is completed and the product is then cooled by cold air.

Recently a separate rewet instantizing plants have been established in Britain; which receive powder for instantizing from various powder factories. It does seem to be a favourable arrangement to have instantizing station with packing plant receiving powder from outside rather than having an instantizer tacked on to a drier (Kreinschmidt, 1969).
2. The physical structure of dried milk

Dried milk exhibits a well pronounced dual physical structure: the structure of the individual particle containing the milk solids and small amounts of moisture and air distributed in the state and arrangement imposed by the drying process used (primary structure), and the structure of the bulk of the dried particles, which represents a typical powder, a system of closely packed solids particles in a gas (secondary structure). There exists quite a diversity of dried milk products, depending upon the variety of raw materials (whole milk, skim milk, buttermilk, cream, ice-cream mix, whey, etc) and methods of drying (roller drying, spray-drying, foam-drying, foam-spray-drying, freeze-drying). Some of the methods are aimed towards procuring a quickly dispersible powder (instantising processes). In addition to taste, flavour and appearance the properties of a dried milk product include dispersibility, free flowability and keeping quality. There are number of requirements as to the properties of dried milk destined for special technical purposes, e.g. dried skim milk for breadmaking; dried whole milk for chocolate and candy manufacture; modified dried milk for baby foods, etc. The storage conditions which may effect the structure and properties comprise the moisture content of the powder, the gas in which it is packed, the packaging material and type of container, storage temperature and time.
(a) Structure of dried milk particle
(primary structure)

The structural elements of a dried milk particle are lactose (normally in amorphous state; in certain instantized powders it is partially in a crystalline state), casein micelles and whey protein particles, fat (partly in globules, partly free) and air (as spherical cells). The protein fat and air are presumably dispersed in a continuous phase of amorphous lactose. However, there are indications of an interconnection between the casein micelles, which thus forms a network. Despite the fact that the powder particles occur in a solid state and the components are confined to fixed sites, chemical reactions affecting the flavour and colour of dried milk may occur at certain places, particularly at the phase interfaces (topochemical reactions).

(i) Shape, appearance and size of particles

The close relationship between the drying process employed and the shape, appearance and size of particles is best revealed under the microscope. Several different microscopical methods have been developed for this purpose, viz., illumination by transmitted ordinary and polarized light, and by incident light, dark ground illumination, fluorescence microscopy, entrapal contrast microscopy and electron microscopy. The specimens for light microscopy are made by mounting the powder in
paraffin oil or various organic liquids. In the case of electron microscopy usually ultramicrotome sections are made.

In the roller drying process the thin wrinkled layer of dried milk removed by the scraper knife from the drum, is broken into small pieces by the screw conveyor, and these are subsequently ground to powder. Milk powder particles consist of flakes (platelets) or irregular, angular shape with wrinkled surfaces and rough edges (Palmer and Dahle, 1922; Washburn, 1922). No air cell or lactose crystals are perceptible within the particles. Their length and width depend on the extent of grinding, whereas the thickness is determined by the thickness of the films. Miyawaki (1923) found the average thickness of the flakes, about 10 μ, the flakes being thicker when precondensed milk is being roller dried.

Characteristic of the particles of spray-dried material is the occurrence of minute air (gas) bubbles in the spherical particles. Duffie and Marshall (1953) attributed this to the following causes: (i) the formation of vapour-impermeable films on the surface of drops, which causes puffing of the particles and is prevented from rapid passage through this film; (ii) the rate of evaporation exceeding the rate of diffusion of dissolved solids back into the particle,
which is followed by concentrating the solids in a spherical shell, (iii) the capillary action of the material on the drop surface drawing liquid and solids to the surface and creating subatmospheric pressures within the particle; (iv) the presence of entrapped air or gases in the feed liquid, which do not escape during drying and spraying.

As to the powder particles of spray dried milk, they are generally spherical and they contain a variable number of air bubbles (Palmer and Dahl, 1922; Tillmans and Strohecker, 1924; Lobmaire, 1960). However, in certain cases there may be encountered elongated and curved, sausage-like particles besides the spherical ones (King, unpublished). According to Palmer and Dahl, the air cells in pressure spray dried powders form a much smaller proportion of the total particle than in centrifugally spray-dried powders, and at times are absent altogether. Also Coulter and Jennes (1943) maintain occasional failure to entrap any air by a quite considerable proportion of spray dried milk particles. The lower air content as judged by the size and the amount of air cells in pressure spray dried powder in comparison with centrifugally spray dried powder was confirmed by Vleeschauwer and Puyvelde (1956) for dried skim milks. In both the cases the air content diminished with increasing degree of precondensation. Hallqvist et al (1949) claimed to have made airless spray-dried
milk, the particles containing no air cells. Zhilov (1966) also obtained under certain conditions of drying centrifugally spray-dried powder in which the particles did not contain air cells but had crater-like indentations on their surfaces.

✓ The surface of the particles of spray-dried milk is usually smooth. However, in certain cases wrinkles and folds have been observed by King in a brand of instant skim milk powder, and by Muller (1964) in slowly dried skim milk. In the photomicrographs by Vleeschauwer and Puyvelde (1956) a fine wrinkling can be seen on the surface of a number of particles in pressure spray-dried skim milk. According to Hayashi (1962) at a higher temperature gradient between the milk droplets and the hot air the particle surface tended to be rougher and the interior more porous. These characteristics seemed also to enhance the agglomeration of powder particles, which is an important feature in the manufacture of instant milk powder.

✓ The particle size in spray drying, according to Duffie and Marshall (1953), is affected by the spraying technique, the properties of the raw material, the solids content of the feed liquid, and the drying conditions.

✓ Hungiker (1949) gave as the particle size range in spray dried milk 10/µ to over 100/µ which is closely
resembling the data of Panasenkov (1952) for spray dried whole milk, skim milk and cream (from 5 to 110, with the most numerous group of 40 diameter). Washburn (1922) considered average diameter of about 150 as the most desirable from the standpoint of easy solubility a particle size below 75/μ being undesirable because of the formation of lumps during the reconstitution process, while too course particles would dissolve slowly. The measurements by Coulter et al (1951) showed considerable variations in the size-distribution of particles depending upon the drying equipment and its operation. Centrifugally sprayed powder showed a much higher percentage of particles in the above 140/μ group and a much lower percentage in the 0-60/μ group than pressure-sprayed powder. The peak in both cases was in the 50-120/μ group. This confirms the earlier findings by Palmer and Dahle (1922) that the particles in pressure-sprayed powders are considerably smaller than in centrifugally sprayed powders.

For measuring the size of the powder particles the powder is dispersed in paraffin oil and certain number of particles measured by means of any eye piece micrometer (Tracy et al, 1951; Jansen et al, 1953; Beckett et al, 1962).

In the foam drying process (puff drying) the
preconcentrated milk is homogenized, cooled rapidly to 13°C, mixed with nitrogen and vacuum dried. The dried mass is crushed through screens and gas packed (Sinnammon et al., 1957; Eskew et al., 1958). The particles in this product are irregular in shape, are very thin and are not powdery. In contact with water they show little tendency to lump.

In the foam spray drying method nitrogen is incorporated into the preconcentrate at high pressure just before spraying and drying (Hanrahan et al., 1962). Under the microscope the particles of such a product (whole milk) appeared as relatively large spheres of dried foam, with a considerable number of the spheres fused into aggregates. There were two types of bubbles in the particles. One type, extremely small and profuse, was located throughout the milk solids; the other, relatively large and less numerous, occupied the interiors of the particles and the spherical protuberances on their surfaces. Presumably the gas dissolved in the concentrate was released on the pressure drop and temperature rise during spraying to form the first type, whereas the second type originated from the dispersed fraction of gas and some of the water vapour evolved during drying. On reconstitution, the gas trapped in the particles is released and rises
to the surface to form a fine grained, persistent froth. The average size of individual powder particles was between 62.9 and 106.0 μm, and the average size of clumps between 140-3 and 420.1 μm.

The particles in freeze dried milk as observed by Nickerson et al (1952) were of irregular shaped and were porous. While whole milk dried directly the product was soft and fluffy, while the precondensed milk gave hard and brittle powder.

A number of instantizing methods made use of moistening the spray dried skim milk and redrying the agglomerated material obtained. In this case under the microscope large agglomerated secondary particles are revealed, which may or may not contain lactose crystals.

(ii) Surface of dried milk particles

The nature of the surface of the particles is of importance in the free flowability of dried milk, a subject which is discussed further below. It is also significant in reconstituting the powder in water, where the first step is the establishment of a contact between the particle and water—the wetting. Coulter et al (1951) noticed, great differences in the rate at which the dried milks were wetted when sprinkled on the water surface. Some powders sank within a few seconds while others floated almost indefinitely. Dried
skim milk wetted more readily than dried whole milk, Nickerson et al. (1952) maintained that freeze-dried whole milk, in general, wetted more slowly than spray-dried milk from the same raw material apparently due to the presence of free fat liberated by the freezing process, while freeze-dried skim milk wetted excellently.

Attempts have been made to enhance the wettability by the use of wetting agents. Kather and Hollender (1955) found that polyoxyethylene sorbitan monooleate (Tween 80) exerted wetting action at a concentration of 0.1% on the reconstituted basis. However, a partial churning of the fat took place with dried whole milk when mechanical stirring was applied. This could be avoided by adding 2 different wetting agents; a combination of 0.06% sorbitan monostearate (Span 60) and 0.04% polyoxyethylene sorbitan monostearate (Tween 60) gave a readily reconstitutable powder with no tendency to churn out. With spray-dried skim milk Gibson and Saithby (1954) observed an increase in the wetting ability on adding polyoxyethylene sorbitan monolaurate (Tween 20) to the pre-condensate at concentration 0.5 to 1.0% on the solids basis.

One of the milk constituents occurring on the surface of particle is the 'free' fraction of milk fat.
Under certain conditions lactose crystals may also appear there. Taneya (1963) using the electron microscope, observed crystals of $\delta$-lactose hydrate (about 2/μ in size) on the surface of particles of pressure spray dried skim milk containing 7% moisture.

- The particles of dried milk may carry an electrical charge on their surface. Manus and Ashworth (1943) noted that spray dried milks made from up to 20% pre-condensed milk were highly charged, and this caused difficulties in their handling during the reconstitution process. Conochie and Wilkinson (1955) found that the dried milk particles moving in an air current were positively charged, retaining their charge for some hours. The charge, however, did not alter the apparent density of powder. A positive charge was also observed by Taneya (1964 c) on pneumatically conveyed spray dried skim. In falling through a chute in an a.c. electric field, the powder particles with a diameter of about 20/μ become mostly positively charged, whereas particles with diameter above 20/μ were neutral. During spraying the droplets of pre-condensate (containing 32.9% water) acquired a positive charge if smaller than 300/μ in diameter and a negative charge if larger.

Schulz (1962 b) followed the electrostatic phenomena in different techniques have been
recently measured by 2 methods: by a permeametric technique making use of gas flow through powder beds (Fox et al. 1963), and by the low temperature (−196°C) absorption of nitrogen by powder particles (Berlin et al. 1964). Some figures of specific surface areas as obtained by these methods are as follows: spray dried skim milk, 0.26 and 0.27 sq.m/gm, respectively; spray dried whole milk, 0.12 and 0.21 sq.m/gm; vacuum foam dried skim milk, 0.08 and 0.16 sq.m/gm; vacuum foam dried whole milk, 0.09 and 0.18 sq.m/gm; foam spray dried whole milk 0.11 and 0.56 sq.m/gm; instantized skim milk, 0.05 and 0.21 sq.m/gm.

Comparison of the data obtained by both methods yielded information on the relative surface roughness or porosity of the powders. Spray-dried milks were less porous than the instantized powders and the vacuum foam-dried and foam spray dried milks.

During the drying process migration of some milk constituents towards the surface of the particles may take place. Holsinger et al. (1964) using a modification of the washing technique of Bockian et al. (1957) who first drew attention to this phenomenon, found that some migration of low molecular weight solutes towards the surface of powder particles occurred during both the spray and foam-drying processes, as well as in some cases of instantizing. Foam dried whole milk particles
were the most uniform in composition. However, no relationship could be established between the orientation of milk constituents within the particles and the dispersibility.

(iii) *State of lactose in dried milk*

Lactose is the major component of dried milk, dried whole milk containing about 32% and dried skim milk about 50% still higher is its content in dried whey where it amounts to about 70%. Therefore it must be expected that at least some properties of lactose will be reflected in the behaviour of dried milk particles. There are two modifications of lactose (alpha and beta), of which the former appears as anhydrous and hydrate, while the later only the anhydrous form has been isolated. These chemical forms may appear either in crystalline or in amorphous, glassy state or they may occur in solution.

In fresh roller and spray-dried powder maintained at a low moisture content, the lactose occurs in an amorphous, glassy state, with alpha and beta modifications in the equilibrium ratio, which gives an indication of the temperature prevailing during the drying process. While the ratio beta:alpha in the glassy lactose is about 1:5, it was found to be about 1:6 in roller and spray-dried powder (Troy and Sharp, 1930).
et al (1957) reported Beta:Alfa ratio of 1:5 for conventional spray-dried skim milk.

The occurrence of the glassy lactose in dried milks was demonstrated by Troy and Sharpe using the humidity equilibrium method, crystal seeing test and examination in the polarizing microscope.

Knoop and Selsnammer (1962) using X-ray diffraction, could distinguish between two different types of dried milks viz, in which the lactose is present in the amorphous state, as α-lactose monohydrate, αβ-lactose, and as a mixture of the last 2 forms.

(iv) State of protein in dried milk

The physico-chemical stability of the protein system (casein and whey proteins) of which there is about 27% in dried whole milk and about 39% in dried skim milk is of the utmost importance in reconstituting or recombining the dried milk for different purposes. Hostettler and Imhof (1963 b) investigated with the electron microscope the casein micelles in reconstituted milk from spray-dried and Calapaj (1962) studied reconstituted milk from roller dried and spray dried. Casein from roller dried milks had coalesced completely, whereas casein spray dried milk still appeared mainly as discrete, more or less spherical particles. The particle size ranged from 10 to 35 μ in the average diameter being as follows fresh milk 119 and
123 m/u, spray-dried milk 143 m/u. According to Realefano and Salome the casein micelle in dried whole milk and skim milk (spray-dried) appeared as more or less spherical bodies.

(v) **State of fat in dried milk**

The amount of fat varies widely in different kinds of dried milks. Dried skim milk contains about 1.5% of fat, dried whole milk about 20-27% and dried cream about 65-75%.

Microscopic examination, especially by fluorescence technique, shows clearly that the fat in dried milk can occur in either a finely emulsified state or in a coalesced, de-emulsified state. On the latter case the membrane around the fat globules has been damaged or entirely removed with the result that the globules are apt to flow together to form 'pools' of fat, Tamao *et al* (1959), Berlin *et al* (1964).

In roller-dried whole milk, although some of the fat is dispersed in smaller globules, the bulk of it appears as large globules and 'pools' of free fat inside and outside the particles. In fresh and properly stored spray-dried milk, the minute fat globules appear uniformly distributed throughout the particle. However, not properly made or stored spray-dried powder shows appreciable amount of free fat, which may spread around the air cell inside the powder particle in the shape of thin layer or it may appear on the periphery and surface of powder particles as patches or drops (King 1986).
The size of the fat globules in particles depends upon the manufacturing methods. They are larger when the milk has not been homogenized before roller-drying; when homogenized, the size is 1 μ (Villanova and Wallars, 1950).

(vi) Water in Dried Milk

To prevent the crystallization of lactose and the other changes which accompany it, the moisture content of dried milk should be below 5.6%. In the American Dry Milk Institute specific Grading Requirements are that the maximum moisture content for different grades of dried whole milk should vary between 2.25-3.0%; for spray dried milk, 3.0 and 4.0%; for roller-dried milk, for dried skim-milk, whether spray or roller dried, the limit is 4.0-5%.

(vii) State of air or gas in dried milk

No gas inclusions are visible under the microscope in the particles of roller dried milk, while the particle of spray dried milk both pressure and centrifugally sprayed contain gas, usually in the form of spherical cells (Palmer and Dahl 1922). Smaller quantities of gas may be absorbed on the powder particles and dissolved in water and in the fat (Coulter & Jenness, 1948). Hallgrist et al. (1949) observed that there is a difference in the appearance
of the air cells depending upon the air content of the powder. In the powders of higher air content the air cells were often situated near the centre of the particle. The $O_2$ in the air cells of spray dried milk particles plays a decisive role in the development of oxidative changes in the milk fat of dried whole milk during prolonged storage. According to Lea et al. (1944) the storage life of dried milk in air at ordinary temperatures and under the best condition is of the order 3 to 7 months for spray-dried whole milk, 6 to 12 months for roller dried whole milk and 12 months or longer for dried skim milk. With packing in an inert gas such as $N_2$, spray dried whole milk keeps at least as well as the roller dried product.

When spray-dried milk is out under vacuum the air slowly diffuses out from the cells. This phenomenon is important in gas packing of dried milk. Lea et al. (1944) observed that while the initial $O_2$ content after evacuation of spray-dried milks was about 140.2%, on standing, the $O_2$ content of the head space gas increased, rapidly at first, and then more and more slowly, until a value of 1.3-5.4% was attained in 5 days. This 'desorption' was, however, very slight in roller dried milk, whose particles
do not occlude air cells; spray dried powders usually desorbed comparatively rapidly.

The increase in the O₂ content of the head space gas in cans of dried whole milk after single evacuation and gassing was found to be roughly proportional to the total volume of entrapped air in the powder particles (Coulter and Jenness 1945).

©(b) Dried milk as a powder (Secondary Structure)

Physical properties of milk powder

Powders are concentrated dispersions of solid particle in a gas. They exhibit properties which in some aspects are similar to the properties of solids, while in other aspects they resemble liquids. The dispersion medium for the dried milk particles is air or, in gas packed powder, nitrogen. The air can exert influence on the powder particles by its oxygen content, which may provoke oxidation reactions in the fat, or it may act as a vehicle for water vapour, which causes crystallization of the lactose. The air space between the particles contribute to the bulkiness of powder, which is of significance in connection with packaging volume, outlay of packaging material and space for storage and for shipping.

Normally, although each individual powder particle is supported by direct contact with other particles, the particles are separate. However, when
caking takes place or in certain instantizing processes, the particles become agglomerated. In compressing the dried milk to blocks or tablets, the strongly agglomerated (sintered) particles form a continuous phase which is interpenetrated by a second continuous phase, the gaseous interstices.

1. Density of dried milk

In dealing with dried milks 2 kinds of density have been distinguished, the apparent or bulk density, particle density of the solids.

The apparent (bulk) density is the weight of the solids per unit volume of the powder, and its reciprocal represents the bulkiness. Sjollema (1963) distinguished between minimum and maximum bulk density, corresponding respectively to the loosest and densest packing of the powder. In the latter case the powder in a conical flask was tapped on the table until no more powder could be added. The bulk density depends upon the shape and inner structure of the powder particles. The roller dried milks, due to the flaky shape and rugged outlines of their particles are less dense, their bulk density varying between about 0.3 to 0.5 g/ml. Bulk density is considerably influenced by the fineness of grinding. The nearly spherical particles of spray dried powders permit a closer packing, the
bulk density being in the range of about 0.6 to 0.6 g/ml. Generally, the apparent density of a powder is the higher the wider the size distribution range with appreciable portion of smaller particles. These are able to fit in to the interstices between the larger ones and fill them. Lea et al (1944) maintained that the bulk density of spray dried whole milk varies appreciably according to the type of plant and degree of precondensation. The finding of Webb and Hufnagel (1945) that the bulk density increases with increasing degree of precondensation was confirmed by Manus and Ashworth (1948). An airless spray dried milk according to Hallqvist et al (1949) had a still higher bulk density—about 0.8 g/ml—not only because of the absence of entrapped air but also because of the relatively uniform size and the smooth surface of the particles. Methods for the estimation of bulk density have been devised by Beckett et al (1962), Harper (1962), Berlin and Pallansch (1963) and Sjollemma (1963).

The particle density is the density of the dried milk particles including the entrapped air regardless of the volume of the voids between the particles. Methods for its estimation have been devised by Beckett et al (1962), Verhoog (1963) and Sjollemma (1963).
The true density or the density of air free milk solids in the dried milk particles have been found to be 1.26 to 1.32 g/ml. for dried whole milk (with 26 to 27% fat) and 1.44 to 1.48 g/ml. for dried skim milk, (Miers and Anderson, 1944) Lea et al. (1944) Lea and Gane, 1946, Berlin and Pallansch, 1963).

Substituting these density values in the expression for the voids in the powder, namely,

\[ \text{Voids} = 1 - \frac{\text{Bulk density}}{\text{True density}} \]

it may be seen that about \( \frac{1}{2} \) to \( \frac{1}{3} \) of the space occupied by the powder is air. This space can be reduced by compression of the powder to blocks or tablets, whereby the oxygen: powder ratio, and the rate of uptake of moisture from the atmosphere are decreased, and the packaging volume and material, as well as the storage space may be appreciably saved.

Kellner (1937) maintains that the pressure should be increased gradually, in order to avoid a rapid closing of the surface pores which would make a further escape of the air impossible. The best results were obtained at 100 to 150 atmosphere pressure, while at higher pressures a leakage of the fat occasionally took place. Mohr and Ritterhoff (1936), using pressures of 60 to 200 Kg/sq.cm, could compress roller
dried whole milk to 1/2 to 1/3 of its original volume. Lea et al. (1944) compressed roller- and spray-dried whole milk to densities of about 1.1 to 1.2 g/ml, without appreciable loss of fat, using pressures of several hundred atmospheres. Thiel (1945) found that much lower pressures are needed to compress roller dried powder to a certain density than spray dried powder.

(11) Free-flowing property of dried milk

The free flowing property, excluding any stickiness and caking, is one of the most important commercial requisites of dried milk (Hunziker, 1949). It has attained particular significance in operating the modern hot drink dispensing machines. Maintaining the free flowability and preventing the caking is also important in a number of other products e.g., dried egg, table salts, powdered sugars, seasoning, fertilizers etc. A usual measure is addition of anti-caking agents to the powders. For fertilizers Silverberg et al. (1957) mention the following factors as influencing caking: size and shape of powder particles, moisture content, length of curing (aging) period, composition and amount and kind of the anti-caking agent (conditioning agent). Watson (1957) points out that the conditioners must exhibit selective adherence to the surface of powder particles (poor
adherence being reflected in excessive dustiness). They also must act as moisture barriers by preferentially absorbing any moisture coming in contact with the powder. Among the conditioners, Watson enumerates Zeol-lex (a sodium aluminium silicate), calcium silicate, magnesium carbonate, tri-calcium phosphate. The agents are used in amounts of about 0.5 to 1%.

For dried milk used in various types of hot drink dispensing machines Linton-Smith and Hansen (1963) found that addition of free flowing agents to certain spray dried dairy products may yield a substantially denser product. In dried whole milk a 0.75% addition of sodium aluminium silicate reduced the volume by 20%. Calcium silicate was also effective, whereas calcium phosphate was not. On the other hand, in sodium caseinate a reduction in volume by 20% was achieved by the addition of 1.5% calcium phosphate whereas the silicates were less effective. Sjollem (1963) studied the effect of tri-calcium phosphate on spray dried whole milk and skim milk, and on an instant whole milk containing about 1% soybean lecithin. A number of other materials (different silica preparations, sodium-Al-silicate, magnesium oxide, aluminium oxide, talc, kieselguhr and activated charcoal) were also tested for their activity
in spray-dried whole milk. Ballschmieter and Heinen (1964) attempted to replace inorganic compounds as free flowing agents by organic materials derived from food-stuffs. The addition of 2% of a specially prepared (dried at 45-50°C) dried whey or 100 to 200 mesh size exhibited a good free flowing effect with roller dried whole milk and with a butter fat powder. The effect was ascribed to existence of lactose crystals of uniform size in the dried whey.

The free flowability of a dried milk may also be enhanced without any addition of conditioning agents. Taneya (1963 a) observed that sorption of moisture unto about 7% by dried skim milk increased its free flowability due to the development of minute α-lactose hydrate crystals (about 2μ in size) on the surface of the particles.

Sjollema (1963) draws attention to the close relationship between the porosity (expressed as percentage of voids) and free flowability. In a very sticky powder the particles adhere to each other, forming very porous structures which are not easily destroyed by tapping or shaking. On the other hand, in a free flowing powder the particles slide easily along each other, filling up a considerable part of
the voids and yielding a less porous structure.
A smaller porosity in turn is associated with higher
bulk density.

Taneya (1963 b) observed 2 types of flow: the
particles flow, where every powder particle moves as
an independent unit, and the block flow, where the
particles agglomerate in to blocks owing to their
cohesion. Dried whole milk showed small block flow
mixed with particle flow, whereas dried skim milk
show particle flow. The flow pattern of instant
dried skim milk was a complicated one.

For the determination of free flowability
either static or dynamic methods have been applied.
Sjollema made use of both types. For the static
method, he used the measurement of the angle of repose—
the maximum angle at which a powder, when piled, will
not slide—according to a modification by Neumall
(1953). The angle of repose is calculated from the
height of the conical pole of powder, which has
flowed freely from a funnel on to a plane surface,
and from the radius of the base of the pile.

For the dynamic measurement of the free flow-
ability, Sjollema used a method by Wouterlood
(unpublished). Here, the rotation speed of a
perspex cylinder half filled with the powder is determined. The powder forms a layer on the wall of the cylinder and rotates with it (so called characteristics speed of the powder). The more free flowing powder, the lower the characteristic speed. Taneya (1963 b) measured the cohesiveness reciprocal of free flowability--by determining the angle of tilt at which the powder slides out from a shallow box, loosely filled to the brim with the powder. For the dynamic measurement of free flowability he used a double cylinder type rotating viscometer. Ballshmieter and Heinen (1964) determined the amount of powder adhering to the wall of a flask of specified dimensions which is rotated at a speed of 30 to 35 r.p.m. for 1 minute.

(iii) Dustability of dried milk

While the free flowability is a desirable property of a dried milk, the dustability--formation of dust during handling of the powder--is a distinct disadvantage. Torrasell (1964) maintains that pressure spray dried milk dust more than centrifugally spray dried milk, and that instant milk forms only little dust. For measuring the dustability he used a method of Anderson et al (1939), in which the powder is dropped through a 2.5 m long tube of
43 mm. internal diameter and the amount of settled powder is determined after a specified fall time at a specified height.

(iv) Reconstitutability

A perusal of literature shows that increasing attention is being given to the reconstitution of dried milk (King, 1966), although reconstitutability of milk powder is a poorly defined and complicated concept. The subject had been quite extensively reviewed by Coulter and Jenness (1964). The task of describing, a method which includes all properties and definitions involved and which applies to different local conditions, therefore, is difficult. The first stage in reconstitution, after the initial contact of water, is wetting of particle surface, (Mohr (1960) and King (1966)), which is followed by water absorption and swelling of protein and solution of soluble constituents (Bokian et al., 1937 and Muers and House, 1962). However, smooth progress of reconstitution process and properties of the product obtained depend upon manufacturing and storage conditions, which affect the nature of the powder particle. Part of the difficulty of rapidly reconstituting milk powder in water lies in the different reactions which occur when milk powder and water are
brought in contact with each other. Solubility, wettability and dispersibility appear to cover most of the reactions involved and suggested as a basis for discussion by Abbot and Waite (1966). These workers define them as:

Wettability:- the rate at which a mass of milk powder is penetrated by quiescent.

Dispersibility:- the degree of separation of wetted powder particles in water.

Solubility:- the degree to which the constituents of milk powder can be brought into solution and stable suspension.

Sinkability:- its rate at which the dried milk sinks into quiescent water. It depends upon the size of particle and surface and particle density of powder.

Abbot and Waite (1966) suggested that the test method should take into account all three characteristics of wettability, dispersibility and solubility, rather than any single characteristic, particularly in relation to domestic use of instant milk powder. However, a wettability test coupled with a solubility measurement, for day to day factory control, both giving results quickly, would provide useful information (Sat Prakash, 1969).
(v) Colour of dried milk

Whole milk, skim milk, dried without overheating to a relatively low moisture content yield a light creamy white powder. Dried milk exposed to the atmosphere in storage suffers more rapid discoloration than gas-packed powder and powder otherwise held in airtight moisture proof containers. Doob, et al (1942) found that in atmosphere with a relative humidity of 20-30%, browning is practically inhibited, but at relative humidities above 30% and up to 55 to 60% the colour increased progressively. Dried skim milk stored at 77°F in storage showed no change in colour but above 86°F they browned rapidly.

Tarassuk and Jack (1943) found that browning does not take place in dried milk and in dried ice-cream mix stored at 40°F or lower, when the moisture content is below 4%.

The literature on browning of milk products has been reviewed by Sharp and Stewart (1946). The browning defect is attributed to two possible reactions, namely, caramelization of lactose and groups of milk protein and the reducing lactose, causing formation of amino-sugar complexes. The fact that high moisture, high storage temperature, alkalinity and packaging in airt-packed containers hasten and intensity the browning is attributed to their accelerating effect on the amino-sugar reaction.
The browning is usually accompanied by other changes also attributed to amino-lactose reaction, such as those that make the powder less palatable and less soluble, that lower the pH and decrease soluble lactose content, that increase titratable acidity and reducing power towards potassium ferricyanide, that cause the absorption of O₂ and production of CO₂.

High moisture in the powder, excessive temperature and long exposure to heat are the chief causes of browning.

3. Microscopic structure

Edler and Dible (1922) explaining the cause of quicker deterioration of spray powders as compared to roller powders observed that:

(a) Spray powder had small spherical granules while the roller dried powder had irregular platelets.

(b) The spherical granules occlude some air at their cores due to the rapid drying of milk spray in large volume of air.

Reifers (1957) said the gas inside the spherical particles was the residual gas. There was found to be a reasonably close correlation between the residual gas volume and the increase in the oxygen content in the interstitial gas when powder cans were gassed with nitrogen and stored for 4 to 14 days.
King (1967) showed that dried milk exhibited a dual structure. The primary structure which referred especially to the internal fine structure of the particles and the secondary structure that referred to the behaviour of the dry products as a bulk. The structure was influenced by the method of manufacture and was reflected in the properties of the dried products.

Particles of spray-dried powder were spherical in shape and contain air cells in the interior. Crystallisation of lactose occurs when moisture is absorbed and a fine network of crystal interstices is distributed throughout the particle. When crystallisation of lactose takes place, the protein particles are expelled to these interstices where the concentration of different substances creates conditions favourable to the destabilisation of proteins and fat. In certain cases the particles are clumped together to bigger agglomerates as in some instant powders.

Verhoog (1963) suggested that 'the amount of entrapped air in milk powder reduced the density of milk powder particles and the powder particles below 1.21 g. per ml. of initial density will float. It is of practical importance since in solubility determination of milk powder the floating fraction should not be ignored.'
Mook and Williams (1966) reported that 'the dispersibility improvement have resulted from the use of processing techniques which reduce the effect of migrated fat on the wettability and increase the porosity of the powder. Powder porosity can be increased by agglomeration procedures and of dryers designed to produce large particles.

Hostettler and Ishof (1936) investigated with the electron microscope the casein micelle in reconstituted milk from spray-dried and freeze-dried powder, and Galanaj (1962) studied reconstituted milk from roller-dried, spray-dried and vacuum-dried powders. Casein from roller dried milks had coalesced completely whereas casein from spray-dried milk still appeared mainly as discrete, more or less spherical particles. The particle size ranged from 40 to 350 μ, the average diameter being as follows: fresh milk 119 and 123 μ for roller dried and 143 μ/μ for spray-dried powder.

Roelofsen and Salome (1961) and Muller (1964) investigated ultramicrotome sections of dried milk particles with electron microscope. In both the studies the particles were fixed with osium oxide (OsO₄). According to them the casein micelle in dried whole milk and skim milk (spray dried) appeared as
more or less spherical bodies. Their diameter ranged between 20 and 30 m/u.

4. Factors affecting the dispersibility of dried milk

The dispersing properties are influenced by the following factors: (i) the chemical composition of dried milk, (ii) the method of manufacture and storage conditions, and (iii) the reconstitution procedure.

The first 2 factors are closely connected with the physical structure of the powder, which in turn is of importance in the reconstitution (N. King 1966).

Effect of composition on dried milk on dispersibility

(1) Milk fat

The fat content of dried milk affects the wettability as well as self dispersion. Ashworth (1955) found that in freshly made fat containing powders, the effect of the fat content on the wettability was only small; 100 samples of medium and high fat content (12.25 and 34%) giving similar high values for the wettability (90-95%). At low storage temperature (1.7 - 7.2°C) these values still remained high. However, storage at 29.4°C caused a considerable deterioration of wettability in medium and high fat powders. Dried skim milk showed wettability value
of about 60% both at low and high storage temperatures, Chakrabôti et al (1956) found a negative correlation between the fat content of powder and its wettability.

Self dispersion diminishes with increase in the fat content of dried milk; for instance, according to Stone et al (1954 a), it was 4.559 g. at a fat content of 1.4%, 1.448 g. at 9.8%, 0.966 g. at 27% fat and 0.527 g. at 39.9% fat. Reinke et al (1960) found that reducing fat content of milk increased the self dispersibility of the powder.

As to the effect of the composition of milk fat on the dispersibility, it appears that with lowering of the melting point the wettability and dispersibility increase. Stone et al (1954 a) found that powders in which the milk fat was replaced by maize oil gave higher self dispersion. Nelson and Winder (1961) observed that with spray dried whole milk the highest sinkability in water at 25°C was always associated with the highest liquid fat content.

Free fat in stored dried whole milk has been considered by Liman and Ashworth (1957) to form an insoluble fat-protein complex, which causes surface scum on the reconstituted milk. The microscopical studies of King (1960) showed that the scum contains numerous clusters and clumps consisting of fat and protein.
(ii) **Milk protein**

Ashworth and Bendixen (1947) reported that 'the physical state of casein is the factor limiting the rate of dispersion of dried milk. King (1962) assumes that the tendency of casein micelle to aggregate is caused by hydrogen bonding between them, which is pronounced with roller dried milks, whereas it is weak with spray dried milks. If the aggregation of casein micelle tends to persist during the reconstitution process, the result is incomplete dispersion, protein flocks appearing on the walls of the containers.'

(iii) **Calcium**

Chakravati et al. (1956) found that partial replacement of Ca with Na (by ion exchange) increased the protein stability, but lower the wettability. Gerlsma (1957) failed to detect any increase in solubility after adding CaCl₂ to skim milk.

(iv) **Lactose**

According to Chakravati et al. (1956) addition of lactose or sucrose to the precondensate improved both protein stability and wettability. After removing the lactose from skim milk by yeast fermentation, Gerlsma (1957) observed a pronounced decrease in solubility of the powder. Addition of 5% lactose to treated skim milk brought the solubility back to the normal level.
(v) Moisture

Ashworth et al. (1954) indicated that the optimum moisture content of dried milk for maximum wettability was about 3.5%. At a moisture content below 2% the powders would be less soluble, while above 4% a decrease in wettability and dispersibility would take place during storage. Abbott and Waite (1963) were of the opinion that in instant-dried skim milk no deterioration in wettability took place during storage at 17°C and 37°C for 24 weeks, so long as the moisture content was below 4%.

5. Effect of manufacturing method and storage condition on dispersibility

For the reconstitution of dried milk for beverage purposes and for the manufacture of different milk products (condensed milk, cottage cheese etc.) a quick and completely dispersible raw material is required. Roller-dried milk, whose solubility behaviour was extensively studied by Weight (1933) is not suitable for these purposes. Presently the most suitable material is produced by the spray process.

(1) Pre-heating

While relatively high pre-heat treatment is necessary for good keeping quality of spray-dried whole milk (an optimum time-temperature of 75.7°C)
for 20 minutes) the pre-heating has very little effect on dispersibility as determined by wettability measurements (Ashworth, 1956). However, any additional heat treatment after condensing the milk does have an adverse affect on the wettability. On the other hand Reinke et al. (1960) found that preheating above 71.1°C for 20 sec reduced the self dispersibility.

(11) **Condensing**

If the milk is not preheated before condensing, the wettability is poor. The optimum solids content according to the Ashworth (1956) lies between 3% and 40%, which substantiates the earlier findings of Ashworth and Bendixen (1944) and Manuel and Ashworth (1948). The latter authors found that powders made from concentrate containing 40% total solids continue to remain 100% soluble after 6 months storage at 7.2°C. Favitova (1959) recommends a total solids content of 43.4% in the concentrate.

With dried milk (both centrifugally or pressure sprayed) Devleschauwer and Van puyvelde (1956) found an increase in the wettability with any increase in the degree of condensation, whereas the solubility was not appreciably affected. Mol and De Varries (1962) also found an increase in wettability with higher total solids content of the pre-condensate.
Vleesheuwer et al. (1957) observed that wettability of spray dried milk increased with preconcentration. No clear effect on solubility or particle size could be established.

(iii) Homogenization

Ashworth (1956) considers the homogenization of precondensed whole milk as the most important step in achieving good wettability. With inadequate homogenization the free fat coats the surface of the dried milk particles, rendering them water-repellent. His experiments showed that homogenization was most effective at fat contents of 1 to 2.5% in the original milk, whereas at fat levels above 3.5%, which corresponds to about 23% fat in the powder, it became increasingly difficult to homogenize efficiently enough to avoid the formation of free fat. Ashworth et al. (1954) also found that in fat range between 12 to 27% (in the powder) the condensing ratio (3:5:1) was more important than the homogenization efficiency for improving the wettability. Passing the homogenized condensate through a centrifugal separator source had improved the wettability.

Reinke et al. (1960) observed that homogenization of milk before condensing or precondensed milk before spray-drying decreased the self dispersability of dried whole milk.
(iv) Spray Drying

According to Ashworth (1955, 1956) variation of the inlet air temperatures between 104.4°C and 100°C seemed to have no effect on wettability. Likewise, variation in outlet temperature between 60 and 32.2°C was without effect. Reinke et al. (1960) noted a trend towards increased self dispersibility when using larger orifice nozzles at lower pressures (formation of larger powder particles).

(v) Cooling of the Powder

Dried whole milk should be cooled down to storage temperature immediately after manufacture to prevent a partial liberation of the fat which would render the particles water-repellent (Hunsiker, 1947). This point is also emphasized by Favorskova (1959).

(vi) Size of Powder Particles

Wilster et al. (1946) maintained that small powder particles favourably influence the reconstitutability of dried whole milk and reduce the scum as well as the film deposit on the container wall.

Swanson (1955), on the other hand, did not find any correlation between the average particle size and dispersibility of dried whole milk. However, the particle size turned out to be an important factor in the dispersing properties of dried skim milk, a
product with particle sizes between 30 and 50/μ exhibiting better wettability and dispersibility than a product with smaller or larger particles.

Gibson andraithby (1954) observed that dried skim milk with a particle size of less than 15/μ was more difficult to reconstitute than on 15–25/μ. Troesch and Willk (1962) found higher wettability with larger particle size. They also drew attention to the shape of the particle surface and the density of particles; the more rugged the surface, the better the wettability, (King, unpublished). While studying the physical analysis of dried milk, Washburn (1922) found that 70 microns in diameter gave maximum solubility. Holler process milk powder particles were ragged looking with waves and wrinkles. The particles in spray process, which were fairly uniform in size, spherical in form.

Panasenkov (1952) found the particle size in 23 samples of spray dried. The average particle size was 35–44.5/μ for dried whole and skim milk. In no case did the diameter of particles exceed 110/μ.

Troesch and Willk (1962) studied the physical characteristics (particle size) structure, specific weight, air volume moisture content etc) of spray dried skim milk using special low pressure nozzles. The powder consisted of crystal like particles of
irregular shape and contained entrapped air, the
density averaged 1.250 g/mL; still better results will
be obtained in a commercial size dryer which would
permit the particle size to be increased.

Kontson and Tassma (1962) found that whole milk
powder produced by using the nitrogen injection techni-
que exhibited a great heterogeneity of particle size.
The large sized particles had the poorest dispersibility,
these particles above 710 μm in average diameter
and constituting 10-35% of total powder mass. A linear
relationship was suggested for the dispersibility of
the powder; amounting to a 4% decrease in dispersibility
per 10% of the coarse fraction present in the
powder.

Karunina (1949) while studying the physical
properties of dried milk, found that density varied
from 0.806 to 0.660 and the size of the particles
3.25 to 125 μm.

(vii) Storage conditions

According to the findings of Ashworth (1956)
the storage temperature was the most important factor
affecting the wettability. Dried whole milk powder
stored at 7.2°C maintained excellent wettability
for as long as 2 years; while the lowest values
were obtained at 29.4°C. At 37.8°C the wettability
increased again. However, these findings did not apply to dried skim milk.

In dried whole milk prepared with the surface-active agents, Hellenda (1955) observed a substantial increase in self dispersion at storage temperatures at or near the melting point of the milk fat, which was attributed to the migration of the surface-active substance to the surface of the particles. Of course, the occurrence of flavours has to be taken into consideration when using higher storage temperatures.

The other important factor which influenced the dispersibility during storage is the moisture content of the powder. Lea and White (1943) found that the solubility of spray dried skim milk with the moisture content of 3.0% and 5.0% remained almost unaltered even after storage for 700 days at 37°C. Powder containing 7.6% moisture, although showing little change in solubility when stored at 20°C, rapidly became insoluble when stored at 28.5°C and 37°C. The decrease in solubility was due to a reaction between the side chains of proteins and the aldehyde groups of lactose, Lea (1943).

(viii) Methods of drying

Spraying drying, moisturing, redrying with various modification are widely employed for instantizing dried skim milk. However, it is unsuitable
for instantizing dried whole milk for which purpose several other methods have been developed i.e. foam drying, foam-spray drying. In this process the primary powder particles being slightly moistened are agglomerated to large secondary clusters possessing a sponge-like structure and subsequently the agglomerated product is redried. Moore et al (1964) observed that the agglomeration while increasing the rate and quality of dispersion has no effect on true solubility.

Bokian et al (1957) are of the opinion that the greatly improved wettability (dispersibility) of instant dried skim milk results mainly from the increased particle size and the distribution of certain constituents in the powder particles. The readily soluble Na, K, Cl and lactose appear to be preferentially oriented on the surface of particles whereas low soluble Ca and protein show preferential orientation towards the centre of the particles. Helsinger et al (1964), however, could not establish any relationship between the orientation of milk constituents within the powder particle and the dispersibility.
6. Effect of preheating temperature on shelf life of dried whole milk.

Manus and Ashworth (1948) found when milk is subjected to four pre-heating temperature i.e. 160°F/80 min, 170°F/30 min, 170°F/10 min and 180°F/10 min, were divided into two parts, one part was concentrated to 20 per cent solids and other to 40 per cent solids, the concentrated milks were homogenized (2000 lb pressure) and spray dried in an pressure type atomizer, the resulting powder were air-packed and examined for palatability at monthly intervals upto 10 months during storage at 45°F and 100°F. The samples, stored at 48°F had shown little deterioration in 10 months, produced from milk preheated at 170°F or 180°F and preconcentrated at 40 per cent solids against 20 per cent solids. All powder samples were deteriorated at 100°F.

Decker et al (1951) found that milk preheated at the temperature of 165°F caused a rapid rise in peroxide value during storage especially after 3 months at 80°F, while preheating at 170°F gave intermediate values. When stored in nitrogen at 35°F all powders had very low peroxide values. It is suggested that the effect of preheating at 170°F in delaying development of oxidized flavour, may be due
to the destruction of an oxidizing enzyme. McDivil, Swanson and Mitter (1960) have studied the effect of forewarming treatment on the flavour of non-fat dry milk. Milk was preheated at 6 temperatures ranging from 195° to 198° F. The samples were rated for degree and type of flavour. Samples heated to 165° F and 175° F were superior to those heated at higher or lower temperatures. Webb and Bell (1942) observed the effect of flash forewarming upon the heat stability of evaporated milk. Forewarming temperatures above 180° F increased heat stability. Jack and Henderson (1942) studied the prevention of flavours in dried milk. They found that when the milk was pasteurised at 170° F for 15 minutes before drying, a marked but not objectionable cooked flavour was caused by this treatment.

Thompson and Kan (1945) found that preheating of the milk at 190° F produced a spray powder that was perfectly palatable even after 12 months of storage in air pack. Mattick (1946) preheated milk at temperatures of 165° and 190° F to 42 per cent total solids, condensed and dried in a hot air chamber using a rotating disc atomizer. He found that there was a close relationship between forewarming temperature
and keeping quality of milk powder. Superiority of high temperature treatments was clearly established. They have larger period of induction and after storage at ordinary temperature for 2½ years, showed no deterioration in quality. Sulphydryl compounds were present in the high temperature samples but not in low ones. It is suggested that the greater resistance to oxidation of the high temperature samples is due to the liberation of these sulphydryl antioxidants and possibly to the more complete destruction of oxidizing enzymes native to milk or produced there in by bacteria. The higher temperature did not significantly reduce the solubility of the powders although it produced cooked flavour.

Tamura et al (1962) have recorded the effect of heating whole milk to (i) 165°F for 30 minutes (ii) 170°F for 6 minutes or (iii) 195°F for 5 second before being vacuum foam dried, packed in air or N₂ containing 1.0 to 0.1 per cent oxygen and stored for 6 months at 90°F. The heat treatment improved the flavour stability of air packed powders, but had little effect on flavour stability of the N₂ packed powders. Cooked flavours and staling of dried products tended to increase as oxidative deterioration during storage was checked by heat treatment in excess of pasteurisation.
Grinod, Jean and Swanson (1963) observed the effect of preheating skim milk on properties of concentrated and dried skim milk, using 30 minutes at 135° - 205°F or 30 seconds at 185° to 205°F. Tests used were pH, solubility index, curd tension, rennet coagulation and packing density. The milk preheated at 155°F for 30 minutes frequently gave indication of less heat treatment than was applied; while higher than expected heat response was obtained with milk heated to 175° - 185°F for 30 minutes. The change in pH and solubility index were not significantly affected by heat treatment.

7. Keeping quality of dried whole milk

Palmer and Dahle (1922) observed that the spray dried powder practically contains a spherical core of air. The presence of air within the granules of powder made by the spray process has an important bearing on the fact that this type of whole milk powder is especially prone to undergo oxidative deterioration.

George (1928) reported that (i) powder prepared from condensed milk showed good keeping quality as compared to heated milk (ii) Homogenization of milk for powder making showed good keeping quality (iii) increase in fat content from 5 to 33 per cent showed progressive improvement in keeping quality.
Schatzel and Clausen (1940) studied the keeping quality of powder stored for 3 months in air, nitrogen and CO₂; storage was at atmospheric (18 to 22°C) and cold rock (10 to 20°C) temperatures. The powder stored under vacuum suffered less deterioration of flavour than air stored powder and showed a much smaller increase of moisture per cent. Use of N₂ and CO₂ showed no advantages over vacuum.

Lea, Holm (1943) reported on the storage of dried skim milk and dried whole milk at 100°F for 6 months. Main conclusions were that the useful life of dried skim milk is not extended by gas packing; that dried whole milk deteriorates much faster than the dried skim milk, if not packed in nitrogen; but that gas-packed spray-dried whole milk generally keeps better than dried skim milk. The quality of dried whole milk can also be improved by packing to 1 per cent oxygen in the head space gas instead of the usual 3 per cent. Same result was observed by Lea and Moran (1943).

Hetrick and Tracy (1949) found that spray dried whole milk occlude less oxygen when the condensed milk had a high solids content.

Greenbank and Wright (1940) while recording the flavour deterioration in 1500 tins of dried whole milk
have established the usefulness of nitrogen packing to reduce the head space oxygen to 3-4 per cent.

Dahle and Palmer (1924) showed that within a temperature range of 4 to 20 °C (39.2 to 68 °F) whole milk powder air-packed in 'Double lid' containers showed scarcely any deterioration after one year, while powders air-packed in identical containers but stored at 37 °C, had suffered pronounced deterioration after 3 months in storage. Same results were obtained by Manuel and Ashworth (1948). Powders made from milk pre-heated at 170 °F/30 minutes and at 180 °F/10 minutes and stored at 45 °F, retained their good palatability even after a storage of 10 months, while the same stored at 100 °F depreciated in palatability during the first month and rapidly developed oxidized flavour.

Lea, Moran and Smith (1943) while studying the effect of gas packing and storage on palatability of whole milk powders concluded that powder containing up to 3 per cent oxygen in the free space gas of can, when stored at (15 °C) 60 °F kept quite well.

Tama, Pollarch and Mucha (1952) studied the factors related to the storage stability of foam dried whole milk. They studied the effect of variation in oxygen level in the packages, heat treatment during
processing, antioxidants, recombination of milk, fat level, degree of lactose crystallization and per cent of free fat on the flavour of foam-dried whole milk during storage. Oxygen level and heat treatment were the most important factors and lauryl gallate was the most effective antioxidant. Products containing fat deteriorated more rapidly than non-fat products. Some authors studied in 1960 the effect of moisture content, storage temperature and oxygen level on flavour stability of foam-dried whole milk. Low heat dried milk containing 5,4,3 or 2 per cent moisture was nitrogen packed or packed in nitrogen containing 1 per cent oxygen or air packed and stored at 30,50,74 or 86°F for 2,4 and 6 months. Moisture had a flavour stabilising effect which was greatest at 34°F and increased with increasing oxygen content. At 80°F or 86°F the flavour stability of high moisture samples was poor.

Junkfar (1962) studied the influence of nitrogen gas packing on the keeping quality of whole milk at different temperature. Dried milk containing 23 per cent fat and 3 per cent moisture was vacuum packed or non vacuum packed and stored for 5 or 12 months at 15 or 35°C. The flavour of vacuum packed milk after 12 months storage at 15°C was still good while that of non vacuum packed milk showed considerable off-flavour. Deterioration was found in all milks stored at 35°C, but was less in the vacuum packed milks.
Scope and Plan of Work
SCOPE AND PLAN OF WORK

The dried milk industry is comparatively of recent origin in India. It is only from the beginning of the Second Plan period about half a dozen factories in private and co-operative sectors have commenced production. The latest available statistics indicate that the total annual production of different categories of dried milks in India works out to about 5,700 tons per year.

Practically the whole of the country's production is derived from buffalo's milk. Although considerable research work has been reported on the difference in the quality of cow milk and buffalo's milk as regard to major and minor constituents, yet there is practically no published literature on dried buffalo's milk. Literature on dried milk published from other countries and review of the previous section clearly indicate that the quality of milk considerably influences the quality of all types of dried milks. There is sufficient reasons, therefore, to expect differences in the quality of milk powder from the cow's milk and buffalo's milk.

Of the different characteristics of dried milk those related to physical characteristics are influenced to a great extent in the various steps involved in the commercial handling of the powder.

It was, therefore, laid as the scope of the present investigation to study the physical characteristics of
differently prepared dried milk as influenced by the conditions of manufacture and storage. The studies were carried out on collected milk of Murrah buffaloes, handled for milk drying at the experimental dairy of the National Dairy Research Institute, Karnal. The different process variants and storage condition studied including the details of analysis carried out are indicated schematically below.

I. Scheme of work for studies on the physical characteristics of roller dried buffalo’s milk.

<table>
<thead>
<tr>
<th>Type of milk</th>
<th>‘Pre-heating’ Temp (°C)</th>
<th>‘Pre-heating’ Time (min)</th>
<th>Concentration Temp (°C), Vacuum of Conc. (mmHg)</th>
<th>Total solids (% in pan)</th>
<th>‘Operating conditions of roller’</th>
<th>Speed of roller (in min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Skimmed milk</td>
<td>80</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>27</td>
<td>60 and 30</td>
</tr>
<tr>
<td></td>
<td>95</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>27</td>
<td>60 and 30</td>
</tr>
<tr>
<td>Standardized milk</td>
<td>80</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>27</td>
<td>60 and 30</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>27</td>
<td>60 and 30</td>
</tr>
<tr>
<td></td>
<td>115</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>27</td>
<td>60 and 30</td>
</tr>
</tbody>
</table>

1) Packaging of powder: Samples of skimmed milk powder were packed in standard polythene bags in 16 kgs lots with an outer covering of paper board and gunny bag. Whole milk powder was air/double gas-packed in standard tagger top tins.

2) Conditions of storage of powder: The samples of skimmed milk powder were stored in a dry ventilated room at
atmospheric temperature. Samples of whole milk powder were stored in incubators at 33°C and at 37°C.

3) Details of observation made:

Skim milk: (i) smell, (ii) colour, (iii) moisture, (iv) pH, (v) bulk density, (vi) solubility, (vii) dispersibility, (viii) wettability and (ix) physical structure (Microscopic).

Whole milk: In addition to the above, oxygen content of head space air, peroxide value and free fats were estimated.

4) Interval of observations: Observations were made immediately after production and at intervals of 2 months.

5) Duration of storage: 12 months.

II. Scheme of work for studies on physical characteristics of spray dried buffalo's milk.

<table>
<thead>
<tr>
<th>Type of milk</th>
<th>Pre-heating Temp.</th>
<th>Pre-heating Time</th>
<th>Concentration</th>
<th>Total solids of ijer</th>
<th>Operating conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>in°C</td>
<td>°C</td>
<td>in °C</td>
<td>in vac</td>
<td>in pan (in)</td>
</tr>
<tr>
<td>Skimmed milk</td>
<td>80</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>45</td>
</tr>
<tr>
<td></td>
<td>95</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>45</td>
</tr>
<tr>
<td></td>
<td>95</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>28, 30, 35, 130</td>
</tr>
<tr>
<td>Standardized milk</td>
<td>80</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>95</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>115</td>
<td>Flash</td>
<td>57.2</td>
<td>63.5</td>
<td>50</td>
</tr>
</tbody>
</table>

Speed of atomizer was maintained uniform at about 25,000 r.p.m.
1) **Packaging of powder**: As indicated in Scheme I.

2) **Conditions of storage of powder**: As described in Scheme I.

3) **Details of observations made**: As described in Scheme I.

4) **Duration of storage**: 12 months.

5) **Interval of observation**: After every two months.

III. Observations on viscosity, size and number of lactose crystals in concentration prior to instant drying.

<table>
<thead>
<tr>
<th>Interval of</th>
<th>Total solids</th>
<th>Holding temp</th>
<th>Observation</th>
<th>to be made on</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>40 and 50</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 hrs</td>
<td>5 and 10</td>
<td></td>
<td>I) Viscosity</td>
<td></td>
</tr>
<tr>
<td>12 hrs</td>
<td></td>
<td>5 and 10</td>
<td>ii) Crystal size</td>
<td></td>
</tr>
<tr>
<td>14 hrs</td>
<td>5 and 10</td>
<td></td>
<td>iii) Number of crystals</td>
<td></td>
</tr>
</tbody>
</table>
IV. Scheme of work for studies on the physical characteristics of instant milk powder from buffalo milk.

<table>
<thead>
<tr>
<th>Type of milk</th>
<th>Pre-heating Temp.</th>
<th>Concentration I (%)</th>
<th>T, S, Speed</th>
<th>Inlet Air Temp.</th>
<th>Atomizer temp. in °C</th>
<th>Cutlet Air Temp. in °C</th>
<th>Effect</th>
<th>Effect</th>
<th>Atomizer (r.p.m.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Skimmed milk</td>
<td>90 Flash</td>
<td>60</td>
<td>50</td>
<td>50</td>
<td>7,000</td>
<td>200</td>
<td>95</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>90 Flash</td>
<td>60</td>
<td>50</td>
<td>50</td>
<td>10,000</td>
<td>200</td>
<td>95</td>
<td></td>
<td></td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>90 Flash</td>
<td>60</td>
<td>50</td>
<td>50</td>
<td>15,000</td>
<td>200</td>
<td>95</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1) Packaging of powder: Instant milk powder was packed in half kg polythene bags.

2) Conditions of storage of powder: Bags were stored under atmospheric conditions of humidity and temperature.

3) Details of observation made: Same as indicated for spray dried skimmed milk.

4) Interval of observation: Same as for other studies.

5) Duration of storage: 6 months.
Studies on physical characteristics of roller dried skimmed milk powder as influenced by conditions of manufacture and storage (cow and buffalo)
SECTION I

Roller Dried Milk Powder (Skimmed Milk Powder).

Present section outlines the procedures followed in the production, storage and testing of roller dried milk powder used for the studies. Cow's and buffalo's milk were used. The cow milk was obtained from the herd at the National Dairy Research Institute, Dairy Farm. The buffalo's milk was obtained through contractors who collected the milk from an area of 30-40 square miles. Normally the cow's milk was chilled and stored and bulk cooled soon after production and kept cold till use. The buffalo's milk was collected in plain 40 litres milk cans and held at atmospheric temperature for 3 to 4 hours before use.

EXPERIMENTAL

Method of production of Roller Dried Skimmed milk powder

a) Selection of Skimmed Milk

600 to 700 litres of fresh, sweet (cow and buffalo's) milk was processed by the market milk section of the experimental dairy for each trial. The raw milk was seperated on the De-Laval triprocess machine set for the minimum fat content in skimmed milk i.e. 0.05 to 0.1 per cent fat. Skimmed milk was subjected to the following tests before condensing.
1) Organoleptic Test.

ii) Alcohol test.

iii) pH.

iv) Fat content.

v) Total solids content.

b) Pre-heating

The milk was heated in a Silkeborg plate heater of 2,500 lbs. per hour capacity with steam as the heating medium. Temperatures of 80°C and 95°C were employed without holding.

c) Condensing

The preheated milk from holding tank was drawn into vacuum pan which had a vacuum of 24 to 25 inches (61 to 63.5 cm). Steam was opened into the coils which were covered by the milk. Vacuum of 24 to 25 inches was kept constant throughout the process of condensing. The total solids content of concentrate was periodically checked by drawing a sample from the outlets provided at the bottom of vacuum pan, till the required total solids 27 ± 0.6 were reached. After that steam supply to the coils was stopped. Vacuum was released and product was pumped to the balance tank.

d) Drying

a) Roller Drying

Pre concentrated milk was dried on Siemens
atmospheric double drum, 18 cast steel roller of 16" diameter.

Rollers were started keeping the non-condensing gas-vent open. Steam was slowly opened to the rollers for heating. After the non-condensible gases and condensate had escaped the cocks were closed and the rollers were allowed to heat up for about 20 minutes to get them uniformly heated up.

The dried powder was collected at first time in trays till the heat equilibrium attended and steam pressure remained constant. When film started to come in uniform on roller without browning, it was received in conveyer and collected in buckets.

The valve of the milk line was opened and knives lowered on the drums, the steam pressure and milk level between the drums was adjusted for steady conditions, adjustment of knives was made to get a uniform dried milk film. The milk introduced into the V-groove between the two rollers was dried by the heat liberated as the steam inside drums condensed. The rollers rotating in the opposite directions picked up a thin film of milk, which after being dried during a part of the rotation (approximate 2.5 seconds) was scraped by an adjustable doctor-blade referred to as the knife. During drying, knives were adjusted for a complete contact with the drum for efficient removal of the
milk-film which could be done with the help of small screws provided along the length of the blade. The flow of milk was adjusted manually for uniform operation by a valve provided on the concentrated milk feed line to the drier. Distance between the drums was adjusted by operating the screws provided on both the ends for shifting one of the drums. The range of adjustment varied between 1 to 1.5 mm. Steam was fed to both the drums through a feed line with a pressure gauge and non-condensate release valve. Steam condensing inside the drums was removed by a system of steam trays.

As the dried milk came down in the form of a thin film, it was allowed to fall in the trays and collected separately as not forming a part of the experimental sample until it was free from burnt particles and moist film and the steam pressure was stationary at the desired mark on guage. Then it was allowed to fall into the conveyors. Two levels of steam pressure viz. 60 P.s.i. and 80 P.s.i. were selected for the present investigation.

60 to 80 P.s.i in rollers was found to be (Wright, 1933) suitable to produce good quality powder as far as moisture content and solubility is concerned.
Milk powder flakes from the conveyors were collected in the buckets and taken to a separate room for the final grinding and packing.

**Sampling**

Skimmed milk powder was filled in gunny bags lined with poly-kraft papers and polythene, immediately after grinding.

**Replications - 3**

**Material and Methods**

Various tests referred in the literature to measure the characteristics of milk and roller dried milk powder were examined and suitable ones have been used with modification in some cases.

1) **Raw Milk**

Raw milk was tested for its initial quality as given below:

a) Organoleptic, alcohol, pH tests were carried out as per the procedure prescribed by I.S.I. 1479 (Part-1) - 1960.

b) Fat content of the milk was determined by Gerber method on lines detailed in IS : 1224 - 1958.

c) Total solids content was determined as per the methods described in IS : 1183 - 1957 using density reading and the fat percentage of milk.

2) **Pre-concentrated Milk**

Pre-concentrated milk was tested for total
solids content by means of a 'Bauch and Lomb' refractrometer and later confirmed by Mondonnier's gravimetric method as reported in 'Laboratory Manual' (Method of analysis of milk and its products) for unsweetened condensed milk.

111) Dried Milk

a) Colour

Evaluation of the colour of milk powder samples was done by Doob (1942) method. The basic chemicals used for making colour standard were as follows:-

- Potassium chromate: 10.0 g.
- Ferric oxide: 0.150 g.
- Manganese: 0.050 g.
- Sodium chloride: 19.00 g.

The composition of standard colour were as follows:

<table>
<thead>
<tr>
<th>Standard No.</th>
<th>Grams of basic mixture compounded with NaCl to make a total 5.00 g.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.000</td>
</tr>
<tr>
<td>1</td>
<td>0.013</td>
</tr>
<tr>
<td>2</td>
<td>0.039</td>
</tr>
<tr>
<td>3</td>
<td>0.078</td>
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<tr>
<td>4</td>
<td>0.147</td>
</tr>
<tr>
<td>5</td>
<td>0.262</td>
</tr>
<tr>
<td>6</td>
<td>0.444</td>
</tr>
<tr>
<td>7</td>
<td>0.715</td>
</tr>
<tr>
<td>8</td>
<td>1.100</td>
</tr>
</tbody>
</table>

But these standards made by above procedure did not suit as these happen to be the standards meant for cow’s milk powder. To suit the present study the
composition of basic colour mixture was modified to:

Potassium chromate 10.00 g.
Sodium chloride 20.00 g.

The quantity of Ferric Chloride and Borrif were kept same as they were in original basic colour mixture.

b) Moisture
Moisture content in dried milk was determined gravimetrically using Mojonnier tester as per method described in 'Laboratory Manual' 'Method of analysis of milk' and its products, i.e.

\[
\text{Percent moisture} = \frac{\text{Loss in weight}}{\text{Weight of sample}} \times 100
\]

c) pH
pH of milk determined by Metrohm pH meter E-350, according to the procedure laid down in IJS 1165 - 1957.

d) Fat
Percent fat in dried milk was tested by Rose-Gottlieb ether extraction method (Mojonnier modification) as detailed in 'Laboratory Manual', 'Methods of analysis of milk and its products'.

e) Bulk density
Bulk density was determined by Sjollem's (1963) method. A 100 ml. graduated cylinder of tared weight was taken. The funnel stem was placed at the mouth of cylinder and the powder was allowed to flow freely to 100 ml. mark. The net weight was obtained and results expressed as g/ml. (Loose density).
To determine the (packed) bulk density the cylinder with the powder was tapped on a rubber mat until the volume was reasonably constant. The volume of powder was read in ml. and density recorded in g/ml.

g) **Solubility Index**

Solubility index was determined by the A.D.M.I. Method (1965) which measures the volume of sediment obtained on centrifugation of a specific volume of reconstituted milk. 10 g. of the skimmed milk powder was reconstituted in 100 ml. of water at 23.9°C by blending in a special mixer for 30 seconds. After standing for a while the foam was removed and 50 ml. of liquid centrifuged in a conical graduated tube for 5 minutes. Most of the supernatent was washed by dispersing it in water and centrifuging again for 5 minutes. The volume of sediment in ml. was designated 'Solubility Index'.

h) **Average particle size by microscopic method**

Average particle size was determined by a modification of microscopic procedure described by Jensen, Swanson and McIntire (1963) and adapted by Beckett et al (1962). Two samples were prepared from each lot of powder by placing about 0.2 g of powder in each of the two vials containing 5 ml of a mineral oil. Two smear were prepared from each sample by placing few drops separately on a slide and covering each carefully with
a cover slip. The smears were examined microscopically with 10x ocular and 20x dry objective. The size of the particles were taken in intervals of 0-1/4, 1/4-1, 1-1 1/4 etc., divisions of the ocular micrometer. As one division was equal to 8.4 micron. The particle diameter were classed in 4.2 micron increments. Fields were selected at random and all particles in a field were classified. One hundred particles were classified per smear. The average particle size (Fpm) determined by the microscope was estimated as follows:

\[ F_{pm} = \frac{\text{F}_{n_i}}{n_i} \text{ micron, where} \]

\[ n_i = \text{each interval of classification.} \]

\[ n_i = \text{number of particles in} \ i\text{th interval.} \]

\[ d_i = \text{diameter midway between the extremes of each interval.} \]

h) Wettability

A very convenient and suitable method developed by Muers and House (1962) for comparing the wettability of instant spray dried milk powder was employed as described below.

The milk powder was floated on to almost completely quiescent surface of a layer of water by spreading the sample over a stretched piece of fabric of suitable mesh and permeability and then gently submerging this small distance under water surface.
The water then penetrated the powder layers and the time for complete wetting judged visually was accurately measured.

The fabric used was Satin drill approximately 225 g/89.m having about 30 threads per cm. in the warp and 20 in the weft. A piece of this cloth measuring about 10 x 10 cm was stretched over one end of the body of a metallic can 6.5 cm diameter and 4.5 cm height open at both ends and was held on with a rubber band. Another open-end can 5 cm. diameter and 7 cm height was placed inside the larger can using rubber corks as spacers to hold it in position centrally on the cloth. A dish 15 x 20 cm was marked at a depth of 2.5 cm, from the bottom and filled with distilled water to this point. A triangle of 0.4 cm. glass rod with sides about 8 cm. long was placed in the dish and served to prevent close contact of the cloth with the bottom of the dish.

With two cans assembled and the cloth resting clean on surface, 2.5 g. of powder was transferred to the inner can and spread over the 5 cm. circle of cloth as evenly as possible with a soft hair brush. The inner can was then removed and the outer can lowered into the dish on to the glass triangle and held in place until the water level in the can ceased to
rise. A stop watch was started at the moment the cloth touched the water and stopped when the powder was completely wetted.

1) Dispersibility

The American Dry Milk Institute adopted the dispersibility method of Stone et al. (1954) to test the instant non-fat dry milk. A.D.M.I. specified the mixing of 52.0 g. of powder with 400.0 ml. of water at 75 °F for 30 seconds using mixer of 192 r.p.m. speed. The A.D.M.I. test has been adapted with modification. The major modification has been the replacement of the Hobert Kitchen mixer and accessories with General Electrical (U.S.A.) Mixer operating at 400 r.p.m. available in the Division because of the non-availability of the prescribed set by A.D.M.I. It was thought to be a possible alternative following the lines of Neff and Morris (1967).

The procedures employed may be described as follows:

The mixer was clamped in a position using a laboratory clamp and stand in such away that it could be raised or lowered, when necessary. One beater was fitted to the mixer having a speed of 400 r.p.m. The mixing bowl had an inner diameter of 7 inches at the top, 5½ inches at the bottom and a height of 5 inches.
The bowl was fitted with an outlet of ½ inch to which was attached a rubber tube closed with a pinch clamp. Below the bowl was put a sieve of 72 mesh (3.3) and a flask with funnel.

Water (400 ml) at 75°F was placed into the bowl and 52 g. of powder was transferred to the surface of water. The mixer was then turned on at 400 r.p.m. and lowered into the bowl. Agitation was continued for 20 seconds after which the beater was left in position in the bowl. The pinch clamp on the outlet of the bowl was opened to release the contents on to the sieve. The screened solution was collected in the flask and diluted to 500 ml. 10 ml portion was transferred to weighed aluminium dish evaporated to dryness in Mojonnier Tester and cooled following the method in 'Laboratory Manual' for total solids determination in milk. The weight of the solids obtained multiplied by 50 gave the dispersibility in gm.

**Justification for methods used to determine the physical characteristics of milk powder**

Rate of dispersion of milk powder during reconstitution is considered the most important factor by which the powder is judged. The first stage of the dispersion in any powdered material is the wetting of the solid particles by the solvent. Milk powders have a very wide range of wettability. Some samples sink readily upon coming in contact with solvent whereas others remain floating on the surface.
Several methods for the estimation of wettability are already in use (Ashworth and Gunthardt 1954; and Baker and Bertok's method, 1959). Murea and House (1962) have proposed a simple method for comparing the wettability of 'Instant' spray dried skimmed milk powders. Wetting times vary from few seconds for good instant powder to several minutes for ordinary spray dried powders. They have also demonstrated that particle size is the most important factor controlling ease of wetting. Being a simple method, for comparative study, this has been used to estimate wettabi-lity of powder (skimmed milk powder).

Sannammar's (1966) method was used for determining the wettability of whole milk powder. It measures the sinkability at the same time as it measure the transmittance of powder particles in solution. This is quick, simple and depends upon the amount of free fat and has been found best to estimate wettability and sinkability of whole milk powder by a single estimation.
### Table I

Production data - Roller dried skimmed milk powder (Cow and Buffalo)

<table>
<thead>
<tr>
<th>Type of milk</th>
<th>Quality of milk</th>
<th>Preheating Treatment</th>
<th>Concentration T.S. % in precon. milk ±0.5</th>
<th>Drying conditions Steam pressures in rollers (Psi)</th>
<th>Speed of rollers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alcohol pH Fat S.N.F. T.S. Temp. ' Time in cm OC</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Cow</td>
<td>-ve</td>
<td>6.80 0.1 8.93 9.03 80 Flash 63.5 57.2 27</td>
<td>60</td>
<td>20</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>± 0.14*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Buffalo</td>
<td>-ve</td>
<td>6.70 0.1 9.61 9.71 80 Flash 63.5 57.2 27</td>
<td>60</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>± 0.23*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cow</td>
<td>-ve</td>
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<td>60</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>± 0.16*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Buffalo</td>
<td>-ve</td>
<td>6.70 0.1 9.52 9.62 95 Flash 63.5 57.2 27</td>
<td>60</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>± 0.18*</td>
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</tbody>
</table>

* Standard error
RESULTS AND DISCUSSION

1) Data obtained on the quality of milk taken for the manufacture of roller skimmed milk powder is presented in Table I.

ii) Initial quality of dried milk in respect of physical characteristics of powder as affected by manufacturing conditions is presented in Table II.

iii) Data obtained on different attributes of roller dried milk powder of cow and buffalo under storage conditions is presented in Table III.

1) Quality of raw milk

From Table I, it is observed that the skimmed milk of cows and buffaloes used for the trials was normal and more or less uniform in composition as judged by percentage of fat and total solids content. 68 per cent alcohol was used for testing the buffalo's milk. At this percentage buffalo's milk samples gave negative results while cow's milk tested at 75 per cent alcohol, were found normal for condensing and drying. There was, however, a slight difference in total solids content of cow's and buffalo's milk.

Production details

A glance at Table I for manufacturing details reveals that the temperatures of pre-heattreatment were
Table II

Effect of different preheating and drying conditions on the physical characteristics of roller dried skimmed milk powder.

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<td></td>
<td>60</td>
<td>80</td>
<td>60</td>
<td>80</td>
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<td>Colour</td>
<td>3</td>
<td>3</td>
<td>3*</td>
<td>3*</td>
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<tr>
<td>Flavour</td>
<td>Normal</td>
<td>Normal</td>
<td>Slightly cooked</td>
<td>Slightly cooked</td>
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<tr>
<td>pH</td>
<td>6.75</td>
<td>6.70</td>
<td>6.65</td>
<td>6.60</td>
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<tr>
<td>Bulk density (gm/ml)</td>
<td>0.325</td>
<td>0.319</td>
<td>0.328</td>
<td>0.331</td>
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<tr>
<td>Solubility index (ml)</td>
<td>11.30</td>
<td>11.90</td>
<td>11.50</td>
<td>12.10</td>
</tr>
<tr>
<td>Wettabiliy (minutes)</td>
<td>5.2</td>
<td>6.1</td>
<td>5.6</td>
<td>6.2</td>
</tr>
<tr>
<td>Particle size (micron)</td>
<td>Range: 20 to 120 microns</td>
<td>Range: 20 to 120 microns</td>
<td>Range: 20 to 120 microns</td>
<td>Range: 20 to 120 microns</td>
</tr>
</tbody>
</table>

SE = Standard error
kept constant at 90° and 95°. The condensing and drying conditions were maintained to be within reasonable limits. The level of total solids in pre-concentrate was kept at 27 ± 0.5 per cent. Two different steam pressures of 60 and 80 P.s.i. were used and the roller has always operated at 30 revolutions/minute.

ii) Physical quality (initial of roller dried skimmed milk powder as influenced by manufacturing conditions.

a) Colour

The initial colour of the product from cow's and buffalo's milk corresponded to 2 and 3. Colour of buffalo's milk powder was almost white while that of powder from cow's milk was yellowish. The temperature of preheating and pressure of steam in the roller had no significant effect on the colour of powder from either types of milk when the intensity of heat treatment was low i.e. 80° C preheating temperature and 60 P.s.i. steam pressure. However, when temperature of preheating increased to 95° C and the pressure in roller was 80 P.s.i., the powder acquired a perceptible brownish colour. The above results are in agreement with findings of Brosely (1945), Mättick et al (1945) and Findlay et al (1946). Manus and Ashworth (1947) have reported that high pre-heating temperatures affected the intensity of colour of powder.
b) **Flavour**

Except in the case of samples corresponding to the high intensity heat treatment, all other samples of powder were observed to give reconstituted milk of normal flavour. In the case of samples corresponding to high pre-heating temperature, the reconstituted milk had a slightly cooked flavour. Similar effects of pre-heating have been reported by other workers Findlay and Mattick (1945).

c) **Moisture**

From data in Table II, it is observed that the moisture content corresponding to low and high steam pressure in cow's and buffalo's milk powder was 3.43 per cent and 3.25 per cent, 3.41 per cent and 3.12 per cent respectively. It is evident that the moisture content of powder from cow's and buffalo's milk was practically same and well within the specification of I.S.I. i.e. 4.0 per cent. It was also observed that the effect of high steam pressure was pronounced since high steam pressure (i.e. 80 P.s.i. as compared to 60 P.s.i.) in the roller gave less moisture content i.e. by 5.5 per cent in case of cow's milk powder and 9.3 per cent in case of buffalo's milk powder. Wright (1932) has reported that when more drastic heat was applied, the moisture in powder decreased as the film was subjected to high temperature during drying.
4) **pH**

The results in Table II show that the pH at 30°C pre-heating temperature and 60 and 80 P.s.i. in roller was 6.75 and 6.70 in the case of cow's milk and 6.7 and 6.65 in the case of buffalo's milk respectively. The pH at 95°C pre-heating temperature of milk and 60 and 80 P.s.i. in roller gave values of 6.65 and 6.60 in case of cow's milk and 6.65 and 6.60 in buffalo milk powder.

From the above results it is seen that the high pre-heating temperature and high steam pressure reduced the pH slightly with either type of milk. The pH of all samples of powder was within the limits prescribed by the I.S.I. Moreover it is seen that the pH in both the cases was found to be almost same, provided the conditions were kept same during drying.

e) **Bulk Density**

A careful look at Table II will show that the range for the values for bulk density of powder from cow's and buffalo's milk overlap (for both loose and packed density measurements). However, the powder from buffalo's milk varied over a wider range as compared to powder from cow's milk. The range for loose and bulk density were 0.319-0.331, 0.381-0.389 gm/ml in cow's milk powder and 0.322-0.335 and 0.385-0.413 gm/ml in case of buffalo's milk powder.
From the above results, it can be said that there was no effect of pre-heating temperature and steam pressure on bulk density of cow's and buffalo's milk powder. Similar results have been reported by Sjollema (1963). The bulk density of roller dried powder is always dependent upon the fineness of grinding. Moreover, the roller dried powder is irregular and flaky in shape, having rugged outlines of particles, which in turn prevent the close packing and thus lower bulk density.

f) **Solubility**

The results in Table 1 show that the solubility index of cow's milk powder at low pre-heating and at low and high steam pressure was 11.3 and 11.9 ml respectively whereas at high pre-heating temperature and low and high steam pressure the values for solubility index were 11.5 and 12.1 ml.

On the other hand, in case of buffalo's milk powder solubility index corresponding to low pre-heating temperature and low and high steam pressure was 12.6 and 13.4 ml respectively, while for high pre-heating temperature and low/high steam pressure, the values were 12.9 and 13.6 ml. respectively.

From the above results, it is revealed that solubility index of buffalo's milk was more by 1.5 ml in all the samples as compared to cow's milk. This difference, however, cannot be considered to be
pronounced as solubility index of either types of powder lie within the specification of A.D.M.I. i.e. 15 ml.

At high pre-heating temperature the solubility index increased by 0.2 ml. in case of cow's milk and by 0.3 ml. in case of buffalo's milk powder. There is, therefore, a slight difference recorded indicative of the detrimental effect of high pre-heating temperature on solubility. However, the increase is not enough to markedly affect the solubility. Mattick et al. (1945) reported that there was minor loss of solubility by pre-heating the milk at 88°C.

The high steam pressure showed definite increase in solubility index in both types of powder i.e. by 0.6 ml. in case of cow's milk powder and by 0.8 ml. in case of buffalo's milk powder. It shows that there was considerable effect of high steam pressure on solubility index of either type of milk powder. The same results have been reported by Wright (1952) with explanation that the drastic application of heat had adverse effect on the solubility of milk powder. It may also be pointed out that notwithstanding the reportedly low heat stability of buffalo's milk, skimmed milk powder produced under standard conditions is equally soluble as the powder from cow's milk. It corresponds to the I.C.I. specifications which are largely based on data collected for cow's milk.
g) Wettability

The data in Table II show that the time to wet one gram powder of cow's milk was 5.2 and 6.1 min. when powder was manufactured at 80°C pre-heating temperature and 60 and 80 P.s.i. steam pressure in roller, while in case of powder manufactured at 95°C pre-heating temperature and 60/80 P.s.i. steam pressure time to wet 1 g. powder was 5.6 and 6.2 min.

On the other hand, in case of buffalo's milk powder, the corresponding timings were 5.3, 6.4 and 5.9, 6.6 minutes.

The above results show that the buffalo's milk powder took little more time i.e. by 0.3 to 0.6 minute to wet 1 g. of powder. Pre-heating temperature had no effect on wettability provided the other conditions of manufacture of powder were the same.

Use of high pressure steam in rollers, increased the time for wetting by 0.4 to 0.8 minutes in both types of milk powder. This increase is the result of the heat denaturation of proteins at the high roller temperatures (Wright, 1932). The wettability of powder depends on the size, shape and appearance of powder. The larger particles, more irregular in shape of roller dried milk are wetted more readily and give the impression of superior dispersibility (Washburn, 1922, Troesch and Milk, 1962).
b) Dispersibility

From data in Table II, it is apparent that the dispersibility of cow's and buffalo's milk powder corresponding to low pre-heating and low steam pressure were 15.32 and 15.13 g. respectively and for high heat treatment the values were 15.09 and 15.23 g. It shows that the dispersibility of cow's and buffalo's milk powder was practically same, provided the conditions were maintained to be constant during manufacture. It is also seen in Table II that pre-heating had no effect on dispersibility.

Dispersibility at higher steam pressures was found to be less by 0.19 to 0.2 g in case of cow's milk powder and by 0.24 to 0.30 g in case of buffalo's milk powder. The tendency for high steam pressure in the rollers is to reduce dispersibility under the present experimental conditions. However, this tendency was not markedly noticeable.

Normally for good dispersibility an important consideration is the total heat treatment on the casein during processing. In roller drying, high heat treatment causes considerably irreversible denaturation of protein which in turn gives considerably less dispersibility as compared to other dried milks, though its particle, shows good wettability at first impression. Wright (1937) had observed similar results and explained that the more denatured the proteins less a-stable is the dispersion.
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<td>(％)</td>
<td>±0.180</td>
<td>±0.261</td>
<td>±0.178</td>
<td>±0.316</td>
<td>±0.234</td>
<td>±0.316</td>
<td>±0.328</td>
<td>±0.239</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SE</td>
<td>±0.130</td>
<td>±0.281</td>
<td>±0.256</td>
<td>±0.296</td>
<td>±0.223</td>
<td>±0.327</td>
<td>±0.283</td>
<td>±0.218</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solubility index (ml)</td>
<td>±0.12</td>
<td>±0.20</td>
<td>±0.10</td>
<td>±0.20</td>
<td>±0.13</td>
<td>±0.10</td>
<td>±0.18</td>
<td>±0.19</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a)</td>
<td>11.3</td>
<td>11.4</td>
<td>11.8</td>
<td>12.3</td>
<td>12.6</td>
<td>12.7</td>
<td>12.9</td>
<td>13.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>b)</td>
<td>11.9</td>
<td>11.9</td>
<td>12.0</td>
<td>12.6</td>
<td>13.4</td>
<td>13.5</td>
<td>13.7</td>
<td>14.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SE</td>
<td>±0.20</td>
<td>±0.16</td>
<td>±0.12</td>
<td>±0.15</td>
<td>±0.16</td>
<td>±0.13</td>
<td>±0.23</td>
<td>±0.23</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

N = Normal; SE = Standard error

a) Data pertaining to powder made from milk preheated to 80°C.
b) Data pertaining to powder made from milk preheated to 95°C.

Temperature during the course of observations varied from 10°C to 35°C.
1) **Particle size**

Particle size was on an average 80 μm and ranged from 20 μm to 120 μm in cow's and buffalo's milk powder. The particle size depends primarily upon the film thickness and grinding. Similar results have been reported by (Miyawaki, 1928). The particle size is the most important factor which affects the bulk density, wettability, sinkability and dispersibility.

**Effect of storage under atmospheric conditions on the physical characteristics of roller dried skimmed milk powder from cow's and buffalo's milk**

a) **Colour**

Observations recorded in Table III indicate that there is a very slight change in the colour of powder from differently pre-heated milk during storage. Powder from cow's milk preheated to 90°C and 95°C recorded at initial value of 3 and 3+ for colour which increased to 3+++ and 3++ corresponding to the pre-heating temperatures after 12 months storage at atmospheric temperature.

Powder from buffalo's milk subjected to similar preheat treatment recorded an initial colour of 2 and 2+ which increased to 2+++ and 2++ corresponding to their preheating temperature in 12 months at atmospheric storage. When moisture content is controlled within the prescribed limit and the condition of storage remains the same, it is the nature of milk which influences colour. Henry et al (1946) reported that the reasons which affect the colour of powder during storage are high moisture and high storage temperature.
EFFECT OF STORAGE UNDER ATMOSPHERIC CONDITIONS ON PHYSICAL CHARACTERISTICS OF ROLLER DRIED SKIMMED MILK POWDER FROM COW'S & BUFFALO'S MILK

- 80°C PRE-HEATING TEMP. & 60 PSI (STEAM PRESSURE)
- 95°C

**FIG. 1**

**COW**

- 80°C
- 95°C

**FIG. 2**

**BUFFALO**

**FIG. 3**

**pH**

- 6.80
- 6.60
- 6.50

**FIG. 4**

**FIG. 5**

**SOLUBILITY INDEX IN ml**

- 11.3
- 11.4
- 11.5
- 11.6
- 11.7
- 11.8

**FIG. 6**

**STORAGE IN MONTHS**

- 0 2 4 6 8 10 12

- 0 2 4 6 8 10 12
b) **Flavour**

In all types of powder, there was no effect on flavour during storage.

c) **Moisture**

It may be observed from data in Table III that the roller dried powder prepared from skimmed cow's milk preheated to 80°C increased in moisture from initial value 3.34 per cent to 3.78 per cent at the end of 12 months storage the overall increase being 15.4 per cent. The same milk when preheated to 98°C and converted into powder by drying on the roller increased moisture content from initial value of 3.33 per cent to final value 3.67 per cent, i.e. an overall increase of 9.6 per cent during the 12 months storage at atmospheric temperature (Figure-1).

Skimmed milk powder from buffalo's milk preheated to 80°C registered an initial moisture value of 3.41 per cent which increased to 3.73 per cent i.e. increase by 9.60 per cent during 12 months. Similar powder from milk preheated to 98°C had an initial moisture content of 3.53 per cent which increased to 3.79 per cent i.e. an overall increase of 7.40 per cent in 12 months (Refer figure-2).
The above observations appear to be indicating that roller skimmed milk powder from buffalo's milk in general absorbs moisture at lower rate during storage under identical conditions of storage. Preheating of milk at lower temperature of 80°C resulted in more rapid increase in moisture than the higher preheating temperature of 95°C in either type of milk.

d) pH

The data in Table III on the pH of the different samples of powder show that initially the pH of roller dried powder from cow's milk corresponding to either type of preheating was slightly more than the pH of corresponding samples of buffalo's milk. However, during storage there was very slight decrease by 0.15 units in the case of all types of powders, neither the type of milk nor the preheating appeared to affect the changes in the pH under the conditions of experiment. Trend of increase in pH has shown in Figures 3 and 4.

e) Solubility Index

The figures of solubility given in Table III and change in solubility index during storage was shown in figures 5 and 6 show that roller dried powder from cow's milk preheated to 80°C increased an initial value of 11.30 ml. to the final value of 12.30 ml. i.e. an overall increase of 1 ml during the period of 12 months. Similar sample of powder from cow's milk preheated to
95°C increased by 0.7 ml. in 12 months from an initial value of 11.9 to final value of 12.6. In case of buffalo's milk roller dried powder corresponding to a pre-heating temperature of 30°C was shown to have an initial solubility index of 12.6 which increased to 13.3 ml. i.e. an overall increase of 1.2 ml. in course of 12 months. Similar samples from buffalo's milk preheated to 95°C had an initial solubility index of 13.4 ml. which increased to 14.1 ml. i.e. an increase of 0.70 ml. in the course of 12 months. It may, therefore, be observed that roller dried skimmed milk powder from cow's milk had lower solubility index than similar samples of buffalo's milk. Between the samples of powder of all the milk, the sample corresponding to a higher pre-heating temperature had higher initial value of solubility index but the rate of increasing solubility index was more for the samples pre-heated at the lower temperature.

The major causes of deterioration in powder as reported by Henry et al. (1946) were high moisture content and higher storage temperature which in turn induce the reaction involving the free amino groups of milk protein and aldehyde group of reducing sugar. In the present study the moisture content of all powder samples were below 4 per cent, so the changes in pH, flavour, colour and solubility index were found to be little.
It may also be pointed out that the test packs for storage studies were made in the winter months with the result that during the first 6 months of storage atmospheric temperature remained low in between 10 to 20°C. During such storage the changes in the attribute have been almost negligible. However, when the temperature increased in the latter half (during summer months) the changes were observed to be more pronounced.
Studies on physical characteristics of spray dried skimmed milk powder as influenced by conditions of manufacture and storage (cow and buffalo)
SECTION II

SPRAY DRIED MILK POWDER
(SKIMMED MILK POWDER)

1. EXPERIMENTAL

a) Selection of skimmed milk

Procedure followed for the selection of skimmed milk was the same as described in Section I.

b) Preheating

Preheating of milk prior to condensing details of preheating including temperature/time factors remained the same as in the case of the earlier studies on roller dried milk.

c) Condensing

Equipment, technique of condensing were the same as in earlier studies except that the total solids in the final concentrate was maintained to be $45 \pm 0.5$ per cent. To study the effect of per cent of total solids in concentrate on characteristics of milk powder, the total solids in concentrate were kept 35, 40, 45 and 50 per cent.

d) Spray drying

Anhydro plant was used having the capacity of 35 kgs of water evaporation per hour. First, the plant was cleaned and then assembled Fig. 1. The atomizer was fixed properly and oil was filled in Wick lubricator. The motors were started in the following order:

1. Exhaust fan,
2. Delivery fan,
FIG. 1. SPRAY DRIER FOR MANUFACTURING CONVENTIONAL MILK POWDER.

1. EXHAUST FAN
2. DELIVERY FAN
3. ATOMIZER
4. OIL BURNER
5. FEED PUMP
6. ROTATING SWEEP
7. STAR WHEEL
8. MILK POWDER Outlet
3. Atomizer,
4. Oil burner,
5. Rotary discharge valve,
6. Stirring device in whirl trap.

feed pump was started first with water when the outlet temperature reached about 100°C. The outlet temperature was kept constant at 95 ± 1°C by controlling the feed rate of water. While the inlet temperature continued to increase to the prefixed limit of 150 or 200°C which was controlled by feed rate of oil in burner and size of feeding nozzle. When the outlet temperature became steady at 95 ± 1°C, feeding of concentrate was started instead of water. The r.p.m. of the atomiser was controlled manually to 25,000 ± 1000. The powder was collected off the cyclone through star wheel in pail kept at the bottom (Fig. 1).

Towards the end of drying when all the concentrate passed through the atomizer, water was switched on to the atomizer which continued running until the outlet temperature was stable. Then the burner was switched off. The feed pump, the delivery fan and atomizer were stopped. The door of the drying chamber was later opened and the atomiser taken off. The cleaning was done by sweeping the walls. During cleaning, the exhaust fan was kept in running condition so that powder could be discharged from the chamber. The drying chamber was then cleaned with hot water after
each set of trials. The feed line was cleaned by recirculation of 0.5 per cent caustic soda at 65°C for 20 minutes and then flushed with hot water.

**Sampling**

Immediately after collecting, the powder was packed in gunny bags lined with 2 ply-kraft papers and polythene.

**Number of replications of**

**I. MATERIALS AND METHODS**

The various tests referred in previous chapter to measure the characteristics of milk and milk powder were used for spray dried skimmed milk powder.

i) **Raw milk**

Quality of raw milk was tested as described in the previous chapter.

ii) **Preconcentrated milk**

Pre-concentrated milk was tested for its total solids by methods given in the previous section.

iii) **Dried milk**

a) **Colour**

The method of estimation of colour using standards remained the same as described in the earlier chapter.

b) **Moisture**

This was determined in the Mohonier bench tester employing the usual precautions.

pH and fat were determined by methods as described in the previous section.
iv) Average particle size by microscopic method

The same method (Jensen et al., 1963) was used as described in the previous section.

v) Average particle density and bulk density of powder

The method followed by Beckett et al. (1962) was used to estimate bulk density and average particle density.

A 100 ml graduated cylinder was filled with about 50 ml of Hexane and stoppered. The volume of hexane ($V_1$) and the weight ($W_1$) were recorded. Powder was added through a funnel to increase the volume about 40 ml. The cylinder was placed on level vibration free surface. After one hour, the volume of powder ($V_2$), the volume of powder and Hexane ($V_3$) and total weight ($W_2$) were recorded. The volumes of the floating portion of the powder were added to $V_3$ when making calculations. Volumes were estimated to 0.5 ml and weights to 0.05 gm.

Estimate was made as follows:

\[
\text{Ave. particle density} = \frac{W_2 - W_1}{V_3 - V_1} \text{ g/ml.}
\]

\[
\text{Bulk density} = \frac{W_2 - W_1}{V_3} \text{ g/ml.}
\]

vi) Solubility

The method of A.D.M.I. used in earlier studies was adopted.
vii) Wettability

The same method of Nurers and House used for roller dried powder was employed for determining the wettability of spray dried milk. This method was preferred because of its scope for comparison of the proper types of spray dried and instant skim milk powders.

viii) Sinkability

The property of sinkability as measured by the method of Bullock and Winder (1966) is evidently related to wettability. Some powders with particles enclosing large air cells do not readily sink after wetting and so give misleading results. Photometric method was used as an alternative for this purpose. Sinkability as measured by the photometric method assumes that good powder would sink immediately giving low transmittance value as suggested by Samhammer (1966). Distilled water 3.5 ml at 20°C was taken in a cuvette and on the surface of water was dusted 10 mg. of powder and per centage of transmittance was measured at 706 milli-micron wave length in a Beckman D.V. Spectrophotometer after 2 and 4 minutes and then after 6 minutes, after giving 6 tapings on the side.

ix) Dispersibility

The A.D.H.I. method has been adopted to determine the dispersibility of powder as described in the previous Section-I.
Table IV
Production data - Spray dried skimmed milk powder (Cow and Buffalo)

<table>
<thead>
<tr>
<th>Type of milk</th>
<th>Quality of milk</th>
<th>Alcohol pH</th>
<th>Fat %</th>
<th>S.N.F. %</th>
<th>T.G. %</th>
<th>Preheating treatment</th>
<th>Concentration Vacuum Temp. °C</th>
<th>T.S.% in precon. milk %</th>
<th>Drying conditions Speed of atomizer</th>
<th>Inlet air Temp. °C</th>
<th>Cutlet air Temp. °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cow</td>
<td>-ve</td>
<td>6.80</td>
<td>0.1</td>
<td>2.88</td>
<td>8.96</td>
<td>Flash</td>
<td>63.5</td>
<td>57.2</td>
<td>45</td>
<td>25000</td>
<td>180</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>± 0.18*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Buffalo</td>
<td>-ve</td>
<td>6.70</td>
<td>0.1</td>
<td>8.92</td>
<td>9.02</td>
<td>Flash</td>
<td>63.5</td>
<td>57.2</td>
<td>45</td>
<td>25000</td>
<td>180</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>± 0.21*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-ve</td>
<td>6.65</td>
<td>0.1</td>
<td>8.74</td>
<td>9.84</td>
<td>Flash</td>
<td>63.5</td>
<td>57.2</td>
<td>45</td>
<td>25000</td>
<td>180</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td>± 0.18</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-ve</td>
<td>6.80</td>
<td>0.1</td>
<td>9.78</td>
<td>9.88</td>
<td>Flash</td>
<td>63.5</td>
<td>57.2</td>
<td>45</td>
<td>25000</td>
<td>180</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>± 0.16</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Standard error
### Table V

Effect of percentage of total solids in concentrate on the physical properties of spray dried skimmed milk powder (Buffalo milk)

<table>
<thead>
<tr>
<th>SL. No.</th>
<th>Particulars</th>
<th>30%</th>
<th>35%</th>
<th>40%</th>
<th>45%</th>
<th>50%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Moisture (%)</td>
<td>0.73</td>
<td>1.16</td>
<td>1.82</td>
<td>2.31</td>
<td>3.25</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.072</td>
<td>±0.050</td>
<td>±0.088</td>
<td>±0.090</td>
<td>±0.07</td>
</tr>
<tr>
<td>2.</td>
<td>Acidity (%)</td>
<td>0.15</td>
<td>0.09</td>
<td>0.13</td>
<td>0.12</td>
<td>0.93</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.014</td>
<td>±0.011</td>
<td>±0.12</td>
<td>±0.007</td>
<td>±0.004</td>
</tr>
<tr>
<td>3.</td>
<td>Fat (%)</td>
<td>0.83</td>
<td>0.92</td>
<td>0.86</td>
<td>0.95</td>
<td>0.93</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.012</td>
<td>±0.021</td>
<td>±0.044</td>
<td>±0.040</td>
<td>±0.020</td>
</tr>
<tr>
<td>4.</td>
<td>Particle size (micron)</td>
<td>15.32</td>
<td>20.21</td>
<td>27.61</td>
<td>31.23</td>
<td>38.33</td>
</tr>
<tr>
<td>5.</td>
<td>Bulk density (gm/ml)</td>
<td>0.351</td>
<td>0.367</td>
<td>0.399</td>
<td>0.427</td>
<td>0.448</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.012</td>
<td>±0.014</td>
<td>±0.003</td>
<td>±0.016</td>
<td>±0.114</td>
</tr>
<tr>
<td>6.</td>
<td>Particle density (gm/ml)</td>
<td>0.93</td>
<td>0.98</td>
<td>1.02</td>
<td>1.04</td>
<td>1.06</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.036</td>
<td>±0.021</td>
<td>±0.020</td>
<td>±0.010</td>
<td>±0.04</td>
</tr>
<tr>
<td>7.</td>
<td>Solubility index (ml)</td>
<td>0.43</td>
<td>0.49</td>
<td>0.63</td>
<td>0.68</td>
<td>0.73</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.030</td>
<td>±0.012</td>
<td>±0.040</td>
<td>±0.016</td>
<td>±0.040</td>
</tr>
<tr>
<td>8.</td>
<td>Wettability (minutes)</td>
<td>38.80</td>
<td>31.20</td>
<td>28.70</td>
<td>20.60</td>
<td>11.20</td>
</tr>
<tr>
<td>9.</td>
<td>Sinkability (transmittance %)</td>
<td>90.20</td>
<td>83.00</td>
<td>77.10</td>
<td>64.00</td>
<td>61.90</td>
</tr>
<tr>
<td></td>
<td>2 mins.</td>
<td>86.00</td>
<td>78.00</td>
<td>72.00</td>
<td>58.20</td>
<td>55.40</td>
</tr>
<tr>
<td></td>
<td>4 mins.</td>
<td>80.00</td>
<td>70.30</td>
<td>60.00</td>
<td>49.50</td>
<td>41.70</td>
</tr>
<tr>
<td></td>
<td>6 mins.</td>
<td>76.36</td>
<td>62.63</td>
<td>52.30</td>
<td>42.88</td>
<td>36.31</td>
</tr>
<tr>
<td>10.</td>
<td>Dispersibility (gm)</td>
<td>3.78</td>
<td>2.36</td>
<td>2.83</td>
<td>3.61</td>
<td>3.31</td>
</tr>
<tr>
<td></td>
<td>SE</td>
<td>±0.84</td>
<td>±0.26</td>
<td>±2.63</td>
<td>±2.30</td>
<td>±0.88</td>
</tr>
</tbody>
</table>

SE = Standard error
RESULTS AND DISCUSSION

The most important properties of dried milks are moisture content, acidity, colour, fat content, solubility index, wettability, sinkability and dispersibility. These properties are considerably influenced by the amount of total solids in concentrate.

Results

i) The data obtained on the effect of percentage of total solids in concentrate on the physical characteristics of spray dried skimmed milk powder of buffalo's milk are presented in Table V.

ii) Data obtained on initial quality of spray dried skimmed milk powder with respect to physical characteristics as influenced by manufacturing conditions are presented in Table VI.

iii) Data obtained on effect of storage under atmospheric conditions on physical characteristics of spray dried skimmed powder from cow's and buffalo's milk are presented in Table VII.

Discussion

i) Moisture

The data in Table V shows that

1) The moisture content varied from 0.73 to 3.28 per cent corresponding to the total solids content of 30 to 50 per cent in pre-concentrate (refer Fig. 7).

2) The increase in moisture content is not proportional to the increase in the total solids content of concentrate.
3) Between the limits of 30 and 50 per cent total solids in concentrate the moisture content of the final product is well within the limits prescribed by Indian Standards Institution.

4) However, since the output of dryer is related to the concentration of total solids, it is always considered desirable to have a solids content of about 50 per cent in concentrate. This recommendation appears to hold good for buffalo's milk also.

5) The thinner the concentrate, the finer the particle size of the powder. Surface of such individual particles naturally contains less moisture. With increase in the per cent total solids in its concentrate, the size of the particles increases and consequently the moisture content is higher.

b) Acidity

From Table V, it is seen that

1) Acidity of different batches of powder manufactured from different percentage of total solids in concentrates was dependent upon the initial quality of milk.

2) The data showed that there was no effect of pre-concentrations on acidity of final product.

3) Acidities of powder corresponding to different levels of pre-concentration did not vary much and were well within the limits of 0.25 per cent (L.A) specified by I.S.I. for good quality of skimmed milk powder.
c) **Fat**

From the data presented, it is apparent that it is the initial fat content of milk which is reflected in the final fat content of the powder. The production data in Table VI shows that the fat content of the milk was 0.1 per cent in all trials and this naturally gave the product a fat content less than 1.0 per cent conforming to the I.S.I. specification of 1.25 per cent.

**d) Particle size**

The data in Table V shows that

1) The particle size of powder has varied from 15.32\(\mu\) to 33.33\(\mu\) corresponding to the total solids of 30 to 50 per cent in pre-concentrate (refer Fig.8).

2) The increase in particle size was almost proportional to the increase in the total solids content. As the total solids in concentrates increased, the particles size also increased. A higher total solids content gives large particles due to increase in viscosity of the concentrate. Particle size is influenced by the milk characteristics, drying conditions and drying system. Centrifugal spray is reported to give larger particles than pressure spray. The average range being 10-250\(\mu\) (Hall and Hedrick, 1960). It is now established fact that the larger the particle size, the more readily will the product be reconstituted. Swanson (1955) reported that the particle size less than 30\(\mu\) will not wet readily because the fine particles are difficult to wet.
and tend to produce lumps which are slow to disperse in water; moreover, it appears that very small particles under 30 μm are the most troublesome because the swelling which occurs in initial contact with water blocks the interstices and hinders the access of more water.

e) Bulk density

From the data in V, it is apparent that

1) The bulk density of powder varied from 0.351 to 0.448 g/ml, corresponding to the total solids of 30 to 50 per cent in the concentrate.

2) As moisture percentage in powder increased the bulk density increased.

3) The increase in bulk density is not proportional to the increase in the total solids but at higher concentration the bulk density is more because of less air content in the particle. Further higher concentration is conducive to larger particles size which in turn gives rise to higher bulk density. Centrifugal spray give trifle lower bulk density because of more air content in particle as compared to pressure spray. Moreover, less uniformity in particle size results in closer packing and thus higher bulk density.

Webb and Huffman (1948) also found that the bulk density increases with increasing degree of pre-concentration.

f) Particle density

A careful look at Table V shows that
EFFECT OF TOTAL SOLIDS CONTENT OF CONCENTRATED MILK ON DIFFERENT PHYSICAL CHARACTERISTICS OF BUFFALO'S SKIMMED MILK POWDER (SPRAY)

FIG. 7

T.S. % OF CONCENTRATION

FIG. 8

T.S. % OF CONCENTRATION

MOISTURE %

PARTICLE SIZE IN µ

FIG. 9

PARTICLE DENSITY IN g/ml

FIG. 10

SOLUBILITY INDEX IN ml

FIG. 11

WETTABILITY IN MINS

FIG. 12

DISPERSBILITY IN gm
1) The particle density of powder particle has varied from 0.93 to 1.08 g/ml, corresponding to total solids of 30 to 50 per cent in the pre-concentrate.

2) As the degree of pre-concentration increased, the particle density also increased. The increase in particle density was found to be proportional to the increase in the total solids content in concentrate. (Refer Fig.9).

Figures for particle density reported in present study were less, compared to those reported by Beckett et al (1962). The average particle density for commercial spray dried non-fat cow milk powder varied from 1.07 to 1.3 g/ml. The reasons for this can be explained by fact that the type of spray was centrifugal which gives more entrapped air than pressure spray products. One of the main processing factors that contributes in particle density is viscosity and air incorporation into the concentrate ahead of drying (Verhoog, 1963; Sjollema, 1963). Hall and Hedrick (1966) have reported that in non-fat dry milk, an increase in moisture content increases the particle density. In the present study, the higher particle density was obtained with large particles corresponding to 50 per cent total solids.

g) Solubility

From data in Table V, it is revealed that

1) The solubility index has varied from 0.43 to 0.73 ml corresponding to total solids of 30 to 50 per cent in the pre-concentrate (refer Fig.10).
2) With an increase in total solids of concentrate there was increase in solubility index. The increase in solubility index was not proportional to the increase in total solids content, but it was slightly higher in case at 40 per cent as compared to 35 per cent total solids. For concentrations higher than 40 per cent solubility index followed regular trend i.e. proportional to the increase in the total solids.

3) Between the limits of 30 and 50 per cent total solids in concentrate, the solubility index of finished product is well within the limits prescribed by A.D.M.I. 1.5 ml.

4) More the total solids in the concentrate, more is the quantum of heat received during condensing, hence more denaturation of proteins resulting in higher solubility index. Solubility index is a cumulative measure of heat damage done to casein and is concentration-dependent and increased semi-logarithmically alongwith the outlet temperature. Above the critical limit of 90°C (Coulter and Jenne, 1964), the solubility index is very high. In the present study, the outlet temperature was 95°C.

h) Wettability

From observations in Table V, it is observed that

1) The wettability in terms of time varied from 33.8 to 11.20 min. corresponding to total solids of 30 and 50 per cent in the concentrate (Fig.11).
2) The decrease in wettability was not proportional to the increase in total solids in concentrate, but it showed more wettability at 45 and 50 per cent total solids.

Observations suggest that the most important factor in wettability is the density of particles (which in turn depends upon the amount of air entrapped and viscosity of concentrate). In general, the denser the particles, the more rapidly, they penetrate the water surface and sink.

In the present study, the test used for measuring wettability gave erratic results between replicates. The main cause of variation in results was the difficulty in spreading the powder quite uniformly over a defined area and such difficulties are generally found with non-free flowing powder as it tends to float after wetting and this may buoy up and delay wetting of part of the powder.

The present findings agree with the findings of Pyne (1961). He has reported that particles of non-fat dry milk should have 40 micron diameter for good wettability. In the present study, the particles of powder were not readily wettable due to their smaller size and poor flowability. A number of workers have reported that increase in the wettability was found with increase in the degree of concentration (Van puyvelde, 1966; Vleesheuwer et al, 1957 and Mol and De Vries, 1962).

1) Sinkability

The data in Table V reveals that
1) As the total solids increase the transmittance of light decreases, the values are from 90.20 to 61.20 at 2 minutes; 80.0, 70.30 and 41.70 after 6 minutes corresponding to the 30 to 50 per cent total solids in the concentrate.

2) The decrease in transmittance was not proportional to the increase in total solids of concentrate. At higher concentration the values decrease rapidly.

The above results reveal that a concentrate of 50 per cent total solids gives larger particles having good sinking property as compared to other concentrations which have given smaller particles associated with poor sinkability. The sinkability always depends upon the particle density and free flowability of powder. In present study, there was wide variation within replicate because of lumpiness of powder particles and poor flowing characteristics.

3) Dispersibility

The data in Table V shows that

1) The dispersibility of powder varied from 16.78 to 36.31 corresponding to total solids of 30 to 50 per cent in the pre-concentration (Fig.12).

2) The dispersibility was maximum corresponding to 50 per cent total solids in the concentrate (i.e. 37.31 gm) as compared to other concentrations. The dispersibility mostly depends upon the particle size and shape of particles. Washburn (1922) reported that the 70/μ particle size was most suitable for good dispersibility.
Table VI

Effect of different preheating and drying conditions on the physical characteristics of spray dried skimmed milk powder

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Cow</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
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<tbody>
<tr>
<td></td>
<td>80°C</td>
<td>95°C</td>
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<td></td>
<td>180°C</td>
<td>200°C</td>
<td>180°C</td>
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<td>200°C</td>
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<tr>
<td>Colour</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>Moisture (%)</td>
<td>2.33</td>
<td>2.12</td>
<td>2.42</td>
<td>2.17</td>
<td>2.36</td>
<td>2.12</td>
<td>2.36</td>
<td>2.15</td>
<td></td>
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<tr>
<td></td>
<td>±0.18±0.13</td>
<td>±0.11±0.21</td>
<td>±0.16±0.14</td>
<td>±0.19±0.21</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>pH</td>
<td>6.80</td>
<td>6.70</td>
<td>6.70</td>
<td>6.70</td>
<td>6.75</td>
<td>6.70</td>
<td>6.70</td>
<td>6.65</td>
<td></td>
</tr>
<tr>
<td>Bulk density (gm/ml)</td>
<td>0.467</td>
<td>0.456</td>
<td>0.469</td>
<td>0.452</td>
<td>0.471</td>
<td>0.464</td>
<td>0.476</td>
<td>0.464</td>
<td></td>
</tr>
<tr>
<td></td>
<td>±0.016±0.012</td>
<td>±0.021±0.016</td>
<td>±0.021±0.013</td>
<td>±0.014±0.013</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Solubility index (al)</td>
<td>0.20</td>
<td>0.42</td>
<td>0.30</td>
<td>0.55</td>
<td>0.60</td>
<td>0.80</td>
<td>0.67</td>
<td>0.85</td>
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</tr>
<tr>
<td></td>
<td>±0.02±0.03</td>
<td>±0.02±0.05</td>
<td>±0.03±0.04</td>
<td>±0.06±0.05</td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Wettability (minutes)</td>
<td>20.40</td>
<td>20.70</td>
<td>20.30</td>
<td>21.40</td>
<td>20.40</td>
<td>21.20</td>
<td>20.80</td>
<td>21.70</td>
<td></td>
</tr>
<tr>
<td>Sinkability 2 mins</td>
<td>61.00</td>
<td>60.50</td>
<td>63.00</td>
<td>62.30</td>
<td>60.00</td>
<td>58.00</td>
<td>61.00</td>
<td>57.00</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(transmittance 4 mins 56.50</td>
<td>58.50</td>
<td>56.30</td>
<td>58.00</td>
<td>57.00</td>
<td>55.00</td>
<td>58.00</td>
<td>51.00</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6 mins 47.50</td>
<td>48.50</td>
<td>45.60</td>
<td>46.30</td>
<td>47.00</td>
<td>48.00</td>
<td>48.00</td>
<td>45.00</td>
<td></td>
</tr>
<tr>
<td>Dispersibility (gm)</td>
<td>36.86</td>
<td>35.50</td>
<td>36.51</td>
<td>35.67</td>
<td>35.74</td>
<td>34.48</td>
<td>35.06</td>
<td>34.26</td>
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</tr>
<tr>
<td></td>
<td>±2.36±2.68</td>
<td>±2.31±2.81</td>
<td>±2.12±2.83</td>
<td>±2.61±2.58</td>
<td></td>
<td></td>
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<tr>
<td>Average</td>
<td>34</td>
<td>31</td>
<td>35</td>
<td>32</td>
<td>35</td>
<td>33</td>
<td>35</td>
<td>32</td>
<td></td>
</tr>
<tr>
<td>Particle size (micron)</td>
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<td></td>
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</tr>
</tbody>
</table>

SE = Standard error
II. Initial quality of spray-dried skimmed milk powder with respect to physical characteristics as influenced by manufacturing conditions

a) Colour

The initial colour of the product from cow's and buffalo's milk powder corresponded to 3 and 2 of the standard. Colour of buffalo's milk powder was almost white while that of cow's milk powder was yellowish. Both temperature of pre-heating and inlet-temperature of air had no effect on the colour of powder from either types of milk when intensity of heat treatment was low. However, when pre-heating temperature was increased to 95°C, the powder acquired a perceptible brownish colour i.e. by 2⁺ and 3⁺.

The high inlet temperature did not affect the colour of powder.

In spray drying the danger of browning as a result of the drying process, is negligible (Hunziker, 1948). Excessive temperatures or time in pre-heating causes a slight increases in colour (Findlay et al 1946).

b) Flavour

From Table VI, it is apparent that except in the case of samples corresponding to high intensity heat treatment, all other samples of powder gave reconstituted milk of normal flavour. In case of samples corresponding to high temperature treatment, the reconstituted milk was having a slight cooked flavour. Webb(1935) has reported similar results. It is now an established fact that the cooked flavour of milk is attributed to the liberation of sulphides
of milk by heat. To avoid cooked flavour, the milk can be heated to comparatively high temperature without objectionably pronounced cooked flavour provided that heating period is practically a few seconds only, and the holding period is very short as reported by Lea et al (1945). High pre-heating temperature is nowadays used for high-heat skimmed milk powder for baking purposes. A high pre-heating temperature helps in complete denaturation of whey protein which in turn imparts good bread baking qualities to the skimmed milk powder.

c) Moisture

Data in Table V reveals that the moisture content of cow's milk powder at low and high inlet temperatures was 2.33 and 2.12; 2.42 and 2.17 respectively. In case of buffalo's milk powder, the moisture content was 2.36, 2.12 and 2.36, 2.15 respectively. It is apparent from above results that the moisture content in each type of powder manufactured at different pre-heating and inlet temperature was well within the specifications of I.S.I., i.e. 4.0 percent. The moisture content at high inlet temperature (200°C) was found low as compared to the powders manufactured at low inlet temperature (180°C). This is the only factor which affected the moisture content of powders. Since outlet temperature was kept constant at 95°C, this is due to the temperature gradient and not the absolute value of inlet temperature. Moisture content of dry milk receives more
attention during the drying operation than the other factors as it affects the rate of several deteriorative changes in milk powder i.e. insolubility, colour, acidity during storage. The high moisture causes rapid physico-chemical changes in the milk powder (Henry et al 1948) while low moisture content i.e. 2 to 3 per cent keeps the powder sufficiently good at 37°C to withstand tropical temperature without serious deterioration and without appreciable loss of solubility (Lea et al 1946). As in the present studies, low moisteres ranging from 2.1 to 2.42, give prolonged keeping quality.

d) pH

The results on pH in Table VI show that the values of pH of cow's milk powder manufactured at low heat treatment and high heat treatment were 6.8, 6.7 and 6.7 and 6.7 respectively. While in case of buffalo's milk powder the pH values were 6.75, 6.70 and 6.70, 6.65 respectively. It shows that the pH value of both powders were found to be same and well within the specification of I.C.I. i.e. 6.6 to 6.7.

The combined effect of high pre-heating temperature and high inlet temperature was found to be negligible on pH value of either type of powder. The decrease in pH value at high temperature was found to be 0.5.

e) Particle size

Table VI reveals that cow's milk preheated at 80°C and dried at 180°C and 200°C inlet air temperature gave average
particles 34/μ and 31/μ corresponding to the inlet air temperature. While in case of milk preheated at 95°C and dried at same temperatures gave average particles size 35/μ and 32/μ respectively.

In case of buffalo's milk preheated at 90°C and dried at 180°C and 200°C inlet air temperatures, the average particle size was found to be 35/μ and 33/μ corresponding to their inlet air temperatures. When milk preheated at 95°C and dried at 180 and 200°C inlet air temperatures, the average particle size were 35.7/μ and 32/μ respectively.

The above results show that average particle size of powder dried at 200°C was less by 3 to 4/μ as compared to powder dried at 180°C. The reason was that the feeding rate at 200°C had to increase in order to maintain the outlet air temperature constant, i.e. 95°C.

f) Bulk Density

From data in Table VI it is observed that the values of bulk density of buffalo's milk powder overlap that of cow’s milk powder. The range of bulk density of buffalo's and cow's milk powder were 0.476 to 0.464 gm/ml and 0.456 to 0.467 gm/ml respectively. The bulk density of buffalo's milk powder was slightly more than that of cow's milk powder by 0.010 to 0.014 gm/ml. Effect of pre-heating on bulk density of powder from either types milk was found to be nil. However, the inlet temperature affected the bulk density in either types of powder. 200°C inlet temperature gave slightly less bulk density as compared to 180°C inlet.
temperature. The reason is that at higher inlet temperatures, the feeding rate had to be increased in order to maintain the outlet temperature at 95°C. Hence, the final product resulted in small particles which affect the enclosed air and subsequently bulk density. Hall and Hedrick (1966), Hanrahan and Konston (1965) reported the bulk density of commercial spray powder as 0.50 to 0.67 g/ml. In the present study low bulk density was noticed because of centrifugal spray used in spray drier which excludes more air in powder particles. Hallquist et al. (1949) observed that the bulk density is influenced principally by the amount of air content.

g) Solubility index

From data in Table VI, it is seen that the solubility index of cow milk, manufactured at low pre-heating 80°C and 180°C and 200°C inlet temperature, was 0.20 and 0.24 ml while in case of buffalo's milk powder values were 0.60 and 0.80 ml respectively. It shows that the solubility index of buffalo's milk powder was higher by 0.40 ml as compared to cow's milk powder. It is also seen from results that the high inlet temperature (200°C) affected the solubility index which increased by 0.20 ml in either type of milk powder.

The solubility index of powder manufactured at 80°C and 98°C pre-heating temperatures and at low inlet temperature was found to be 0.20 and 0.30 ml, respectively, in case of cow's milk and in case of buffalo's milk, the values
were 0.60 and 0.67 ml. respectively. It is apparent that the solubility index was influenced mainly by the species character of milk used and not to any appreciable degree by the pre-heating temperature.

In spray drying, the conversion from the pr-conden-
sed to the dried powder is almost instantaneous, the danger of heat damage and loss of protein solubility during spray drying therefore, is practically non-existent.

b) Wettability

Results in Table VI show that the time to wet in case of Cow's milk powder was found to be 20.40 and 20.70 minutes for one gram powder while in case of buffalo's milk powder, the values were 20.40 and 21.25 minutes when manufactured at 180° and 200°C inlet temperature and 80°C pre-heating temperature. It shows that the wettability of either type of powder was almost same.

The values of wettability of powder when pre-heated at 80°C and 95°C and at inlet temperature of 180°C were 20.40 and 20.30 minutes in case of cow's milk powder while in case of buffalo's milk the values were 20.40 and 20.30 minutes. It reveals that the pre-heating temperature had no effect on wettability of either type of powder (Ashworth 1959). The wettability of powder produced at 180°C and 200°C inlet temperature was found to be 20.40 and 20.30, and 20.70 and 21.40 minutes in case of cow's milk powder respectively and in case of buffalo's milk the wettability was 20.40, 20.30 and 21.20 and 21.70
minutes respectively. There appears to be slight influence of high inlet temperature on wettability of both types of powder.

The major factors which affect the wettability of powder are however, particle size, amount of air enclosed and particle density.

1) **Dispersibility**

From results in Table VI it may be seen that the dispersibility of powder made from cow’s milk was 35.86, 35.50, 36.51 and 35.67 g. corresponding to the two temperatures of pre-heating and the two temperatures for the inlet air. In case of buffalo’s milk powder, the dispersibility was 35.74, 34.43, 35.06 and 34.28 g. corresponding to similar variations in preheating temperature and inlet air. The dispersibility of cow’s milk powder was found more i.e. by about 1.2 g on an average as compared to buffalo’s milk powder.

The dispersibility of cow’s milk powder made from 80°C and 95°C preheated milk, was found to be 36.86 and 36.51 g and of buffalo’s milk powder was 35.74 and 35.06 g dried at 120°C. It reveals that the pre-heating temperature had very little effect on dispersibility of either type of powders (Ashworth, 1956). The dispersibility of powder dried at 120 and 200°C inlet temperatures was 35.86 and 35.50 g respectively, in case of cow’s milk powder and 35.74 and 34.43 respectively in case of buffalo’s milk.
Table VII

Effect of storage under atmospheric conditions on physical characteristics of spray dried skimmed milk powder from cow's and buffalo's milk

<table>
<thead>
<tr>
<th>Particulars</th>
<th>Cow</th>
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<tr>
<td></td>
<td>Period of storage in months</td>
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<td>4</td>
<td>8</td>
<td>12</td>
<td>0</td>
<td>4</td>
<td>8</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>Colour</td>
<td>a) 3</td>
<td>3</td>
<td>3+</td>
<td>3++</td>
<td>3+++</td>
<td>2</td>
<td>2+</td>
<td>2+</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>b) 3+</td>
<td>3+</td>
<td>3+</td>
<td>3++</td>
<td>3+++</td>
<td>2+</td>
<td>2+</td>
<td>2+</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Flavour</td>
<td>a) N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td></td>
</tr>
<tr>
<td></td>
<td>b) N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td>a) 6.80</td>
<td>6.80</td>
<td>6.70</td>
<td>6.60</td>
<td>6.60</td>
<td>6.75</td>
<td>6.70</td>
<td>6.65</td>
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<tr>
<td></td>
<td>b) 6.70</td>
<td>6.70</td>
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<td>6.60</td>
<td>6.60</td>
<td>6.70</td>
<td>6.70</td>
<td>6.65</td>
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<tr>
<td>Moisture (%)</td>
<td>a) 2.33</td>
<td>2.43</td>
<td>2.52</td>
<td>2.70</td>
<td>2.70</td>
<td>2.36</td>
<td>2.58</td>
<td>2.61</td>
<td>2.63</td>
<td></td>
</tr>
<tr>
<td></td>
<td>b) 2.52</td>
<td>2.51</td>
<td>2.58</td>
<td>2.89</td>
<td>2.89</td>
<td>2.36</td>
<td>2.41</td>
<td>2.48</td>
<td>2.63</td>
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</tr>
<tr>
<td>Solubility</td>
<td>a) 0.20</td>
<td>0.40</td>
<td>0.85</td>
<td>1.50</td>
<td>1.50</td>
<td>0.60</td>
<td>0.75</td>
<td>1.50</td>
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<tr>
<td>index (ml)</td>
<td>b) 0.30</td>
<td>0.50</td>
<td>1.10</td>
<td>1.60</td>
<td>1.60</td>
<td>0.80</td>
<td>0.90</td>
<td>1.20</td>
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<tr>
<td>Wettnessability</td>
<td>a) 20.30</td>
<td>21.60</td>
<td>22.20</td>
<td>22.80</td>
<td>22.80</td>
<td>20.60</td>
<td>21.30</td>
<td>22.60</td>
<td>23.30</td>
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<tr>
<td>(minutes)</td>
<td>b) 20.80</td>
<td>21.90</td>
<td>22.50</td>
<td>22.60</td>
<td>22.60</td>
<td>20.30</td>
<td>21.20</td>
<td>22.20</td>
<td>23.00</td>
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<tr>
<td>Dispersibility</td>
<td>a) 36.86</td>
<td>35.92</td>
<td>34.58</td>
<td>33.52</td>
<td>33.52</td>
<td>35.74</td>
<td>34.56</td>
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<tr>
<td>(gm)</td>
<td>b) 35.51</td>
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<td>32.25</td>
<td>31.48</td>
<td>33.79</td>
<td>31.73</td>
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</table>

N = Normal; SE = Standard error

a) Data pertaining to powder made from milk preheated to 80°C.

b) Data pertaining to powder made from milk preheated to 95°C.

Temperature during the course of observations varied from 10°C to 35°C.
powder. The dispersibility at higher inlet temperature was found to be less by 1.3 g. with either type of powder. The reason may be that the rate of feeding was increased at higher inlet temperature at 95°C which in turn gave small particle size and slightly less solubility.

III. Effect of storage under atmospheric conditions on physical characteristics of spray-dried skimmed milk powder from cow's and buffalo's

a) Colour

The data on the colour of differently prepared powder during storage are presented in Table VII. It may be observed from these data that there is very slight change in colour of powder from differently prepared milk during storage. Powder from cow's milk preheated to 90 and 95°C recorded an initial value of 3 and 3⁺ respectively which increased to 3++++ and 3+++ after 12 months storage at atmospheric temperature. Powder from buffalo's milk subjected to similar preheating temperatures which increased to 3⁺ and 3 in 12 months storage at atmospheric temperature.

It may, therefore, be concluded that the high temperature showed initial colour 3⁺ in cow's milk powder and 2⁺ in buffalo's milk powder but at the end of storage period, change in colour was less as compared to low preheating temperature of either types of milk powder. When moisture is controlled to be within prescribed limit and the conditions of storage remains same, it is the nature of milk which influenced the colour (Henry et al, 1945).
EFFECT OF STORAGE UNDER ATMOSPHERIC CONDITIONS ON PHYSICAL CHARACTERISTICS OF SPRAY DRIED SKIMMED MILK POWDER FROM COWS & BUFFALO'S MILK

- 80°C Pre-heating temp. & 180°C Inlet temp.
- 95°C COW
- 80°C COW
- 95°C COW
- 95°C BUFFALO

**Fig. 13**

**Fig. 14**

**Fig. 15**

**Fig. 16**

**Fig. 17**

**Fig. 18**
b) **Flavour**

In all types of powder there was no effect on the flavour caused by storage under the controlled conditions.

c) **Moisture**

Observations on the moisture content of differently prepared powder during the storage presented in Table VII and Fig. 13 & 14 indicate that the spray dried skimmed milk powder prepared from skimmed cow's milk preheated at 80°C increased from 2.33 to 2.70 percent at the end of 12 months storage, the overall increase being 15.9 per cent. Same milk when preheated at 95°C, showed an increase in moisture content from initial value of 2.42 to 2.84 percent i.e. an overall increase of 17.3 per cent during 12 months storage.

Skimmed milk powder from buffalo's milk preheated to 80°C registered an initial value for moisture content 2.36 per cent which increased to 2.34 per cent i.e. by 19.5 per cent. Powder from milk pre-heated to 95°C had initial moisture content of 2.21 per cent which increased to 2.63 per cent i.e. an overall increase of 17.4 per cent in 12 months.

d) **pH**

The data in Table VII on the pH of different samples of powder show that initially pH of spray dried powder from cow's milk corresponding to either type of pre-heating was slightly more than the pH of corresponding samples of buffalo's milk powder. During storage, there
was decreased by .2 units in the samples prepared from milk preheated at 85°C and 0.1 units in samples prepared from milk preheated at 95°C in either types of milk powder (Refer Fig. 15 & 16).

It appears from above observations that the sample manufactured from milk preheated at 95°C showed less decrease in pH in either types of milk powder.

e) Solubility Index

The figures for solubility index given in Table VII and Fig. 17 & 18 show that spray dried powder from cow’s milk preheated to 80°C increased from an initial value of 0.20 ml. to the final value of 1.5 ml i.e. an overall increase of 1.3 ml. during the period of 12 months.

Similar samples of powder from cow’s milk preheated to 95°C increased by 1.3 ml. in 12 months from initial value of 0.30 ml. to the final value of 1.6 ml.

In case of spray dried powder from buffalo’s milk corresponding to the preheating temperature at 80°C was shown to have an initial solubility index of 0.60 ml. which increased to 2.20 ml. i.e. 1.6 ml in course of 12 months.

Similar samples from buffalo’s milk preheated at 95°C had an initial value of solubility index of 0.80 ml. which increased to 2.30 ml. i.e. an increase of 1.5 ml in course of 12 months.

It may, therefore, be observed that (i) spray dried skimmed milk powder from cow’s milk had lower solubility
EFFECT OF STORAGE UNDER ATMOSPHERIC CONDITIONS ON PHYSICAL CHARACTERISTICS OF SPRAY DRIED SKIMMED MILK POWDER FROM COW'S AND BUFFALO'S MILK.

**COW**

**BUFFALO**

**FIG. 19**

**FIG. 20**

**FIG. 21**

**FIG. 22**

- 37.08
- 34.98
- 32.88
- 30.78

- 23.30
- 22.30
- 21.30
- 20.30

- 80°C PRE-HEATING TEMP. & 180°C INLET TEMP.
- 95°C

- COW

- BUFFALO

ATMOSPHERIC TEMP. -10 to 3°C
index than similar samples of buffalo's milk powder (ii) high preheating temperature had a slightly higher initial value for solubility index in either types of milk but the rate of increase in the solubility index was more or less the same for either type of milk.

f) *Wettability*

It is apparent from data of wettability in Table VII that the spray dried skimmed milk powder manufactured from cow's milk preheated to 80 °C had increased from 20.30 minutes to final 22.90 minutes i.e. an overall increase in time of 2.60 minutes in course of 12 months period. Similar samples of powder from cow's milk preheated to 95 °C increased from 20.80 minutes to 22.60 minutes i.e. an overall increase of 1.80 minutes in the same period.

Powder from buffalo milk preheated at 80 °C showed an increase from 20.60 to 23.0 minutes i.e. by 2.4 minutes during storage for 12 months. Similar samples of powder manufactured from milk preheated at 95 °C increased from 20.30 to 23.0 i.e. by 2.7 minutes.

The above observations show that the wettability had increased more in buffalo's milk powder as compared to cow's milk powder.

g) *Dispersibility*

The data on dispersibility in Table VII show that the skimmed milk powder from cow's milk preheated to 80 °C decreased in dispersibility from 36.86 gm to 33.52 gm i.e. by 3.34gm during the 12 months storage. Similar sample
of powder manufactured from milk preheated at 95°C decreased in dispersibility from 35.80 g to 32.25 g i.e. an overall decrease of 3.55 g. In case of spray dried skimmed powder from buffalo's milk preheated to 90°C dispersibility decreased from an initial value of 35.74 g to the final value of 31.23 g i.e. an overall decrease of 4.51 g. Similar sample of powder from milk preheated at 95°C decreased by 3.70 g in 12 months from initial value of 34.48 to the final value of 30.78 g.

It appears from the above observations that the decrease in dispersibility was steady in either samples of powder during 12 months storage (Fig. 21 & 22). However, buffalo's milk powder showed slightly more decrease in dispersibility during storage as compared to cow's milk powder. The samples corresponding to a higher preheating temperature had a low initial value for dispersibility but the rate of decrease in dispersibility was more for the samples preheated at the lower temperature.

Ashworth et al (1954) indicated that the optimum moisture content of dried milk for maximum wettability was above 3.4 per cent and above 4 per cent, it showed decrease in wettability and dispersibility during storage period. In present study the moisture percent of all samples of powder was below 4 per cent.

Lea and White (1948) found that the solubility of spray dried skim milk with moisture content of 3.0
per cent and 5.0 per cent remained almost unaltered even
after storage for 700 days at 37 °C. Powder containing
7.6 per cent moisture showed rapid decrease in solubility.
The decrease in solubility was due to a reaction between
the side chains of proteins and aldehyde groups of lactose.

The observations of Lea and White (1943) correspond
to the observations made in the present studies when one
falls into considerations that increases in solubility
and moisture content are only marginal and that the
storage in the present studies was in poly-packs as
against the hermetically sealed tins used in Lea's
studies.
Section - III

Production of skimmed instant buffalo's milk powder
SECTION III

INSTANT MILK POWDER

1. Experimental

Present section outlines the procedures used to manufacture the instant milk powder from buffalo's milk. The buffalo milk was procured from contractors who collect milk from an area of 30-40 square miles around Karnal.

Before selecting the raw milk, it was subjected to the following tests:

i) Organoleptic test,
ii) Alcohol test,
iii) Acidity,
iv) Fat content and
v) Total solids.

Preheating

Procedure and equipment used remained the same as in earlier studies. Powder was made corresponding to the low pre-heating temperature of 90°C as a low temperature is recommended for instant milk (Knipschildt, 1969).

Condensing

Preheated milk was condensed to desired level of total solids in a volma double effect, falling-film evaporator operating at 55 and 65 cm. vacuum in 1st and 2nd effect respectively. The concentrate was periodically checked for total solids.
Holding of pre-concentrated milk prior to instant drying

A preliminary study was made to observe the initial viscosity of milk precondensed to 40 and 50 per cent levels of total solids by means of laboratory rotary evaporator operating at 22-24 inches of vacuum. Samples were subsequently stored at 5 and 10°C for 10, 12 and 14 hours. The pre-concentrated milk was held in stainless steel vessels with stirring facilities. The effect of above mentioned factors was studied on the viscosity, number of lactose crystals and size of crystals. This study was felt essential to fix the suitable total solids in concentrate, time of holding for maximum crystallization in order to give good instant powder prior to actual production of 'instant milk powder'. The most suitable concentrate after condensing (i.e. 50%) was kept for 12-14 hours and agitated in refrigerated bulk milk storage tank until drying. The concentrate meant for instant powder production was stored at 5°C for about 12-14 hours while for normal powder production, the concentrate was dried immediately on a spray drier.

Instant non-fat dried milk powder

Except for the change in the discharge point of the milk powder and the recirculation of the fine particles recovered from the outgoing air in the cyclone
as shown in figure II and flow diagram 2, the operating procedure followed were same as described in previous chapter. The percentage of total solids in the concentrate was 50 ± 0.5% and it was held at 5°C for 12-14 hours prior to drying. A preliminary trial at about 25,000 r.p.m. showed that high proportion of powder particles went along with the air and got separated into the cyclone. Very small fraction settled at the bottom of the drier. This indicated the need for lower r.p.m. of atomization. The r.p.m. of the atomizer was, therefore, changed to 7,000; 10,000 and 15,000 to study the suitable r.p.m. for instant powder and maintained within ± 1000 r.p.m. during all trials.

Packaging

Instant milk powder was collected in polythene bag (poly-kraft). Half kg lots in polythene bags were separately packed immediately after manufacture and stored at room temperature (28-30°C).

B. Material and Methods

Various tests referred in the literature to measure the characteristics of milk and milk powder of Roller dried, Spray dried and Instant types were examined and suitable ones have been used with modification wherever necessary.

1) Raw milk

Raw milk was tested for its initial quantities as given below:
a) Organoleptic, alcohol and acidity tests were carried out as per the procedure given in I.S. 1479 (Part-I) - 1960.

b) Total solids content was determined as per the methods described in I.S. 1183-1957 using density reading and the fat percentage.

c) Fat content of the milk was determined by Gerber method on lines detailed in I.S. 124 - 1958.

2) Precondensed milk

a) Total solids

Precondensed milk was tested for total solids content by means of a 'Bausch and Lomb' Refractometer and later confirmed by Kojonnier's gravimetric method as reported in 'Laboratory Manual: Methods of analysis of milk and its products' for unsweetened condensed milk.

b) Viscosity determination

Viscosity was determined every time at 20°C using a Kromer viscometer as per the details given by Manche Gowda (1967). The fall time in seconds of ball at 20°C was noted and dynamic viscosity in centipoise obtained using the following formula:

\[ n = \frac{F(k - kf)}{K} \]

where

\[ n = \text{Dynamic viscosity of the sample in centipoise.} \]
F = Fall time of ball in seconds.
Sk = Specific gravity of ball.
Sf = Specific gravity of liquid.
K = Ball constant.

c) **Microscopic method of measuring the number and size of lactose crystals**

The use of microscope in detecting crystallized lactose in cooled concentrate has been somewhat limited due to the difficulty in the preparation of cold specimen to prevent the solution of very small crystals.

The method was modified following the suggestion by Whitker (1933) for working at room temperature. This method consisted of preparation of saturated lactose solution at 35-37°C and filtering it carefully at that temperature to remove any crystals which may be present, and allowing the filtrate to cool to room temperature. To this solution was then added water soluble nigrosin dye at the rate of five per cent so as to colour the background of the field. A drop of this supersaturated solution with nigrosin was placed on a 18 mm square area marked on a glass slide and weighed. A small sample of the cold milk concentrate was introduced into the lactose-nigrosin solution on the slide and the slide was weighed again. The difference gave the weight of milk concentrate taken. The weight of the concentrate was adjusted so as to get 20-200 lactose per field. The sample of concentrate plus lactose-nigrosin solution
was stirred to form a uniform mixture and spread over the specified area. The slide was then covered with a cover glass and the preparation viewed through microscope using low power objective. The lactose crystals in the concentrate did not dissolve at room temperature because they were suspended in a saturated lactose solution. The dilution effect was counter balanced by having the lactose solution saturated at a temperature slightly higher than at which the work was done. The size of the lactose crystals was measured with an ocular micrometer. About 300 crystals per sample and all crystals in a field were classified. The average number of crystals per field was also noted. The fields were selected at random.

The average size was estimated by using the formula:

\[ \text{Cs} = \frac{\text{Fdi}}{\text{fi}} \] Micron, where

\( \text{Cs} \) = Average size of crystal

\( i \) = Each interval of classification

\( fi \) = Number of crystals in the \( i \)th field

\( di \) = size midway between the extremes of each interval.

The number of the lactose crystals per g. of the concentrate was determined by using the formula given below (Chalmers, 1962).
Number of lactose crystals per g. of concentrate = \frac{18 \times 18 \times \text{ave. no. of crystals per field}}{x \times (\text{radius of field in mm})^2 \times \text{wt. of sample}}.

**Average particle size by Microscopic method**

Method of Jensen et al. (1953) has been used as described in Section 2.

**Average particle density and bulk density of powder**

Method of Beckett et al. (1962) was used to determine average particle size and bulk density. The method is described in Section 2.

**Solubility**

The method of A.D.W.I. used and described in Section 2.

**Adhesibility**

The same method was used as described in Section 2.

**Wettability**

A method of Mayers and House, was used to compare the wettability in time of conventional spray powder and instant milk powder.

**Dispersibility**

An A.D.W.I. method with some modification to determine dispersibility of instant milk powder, described in Section 2 was used.
Table VIII
Viscosity size and number of lactose crystals in buffalo skin milk concentrate held at varying temperatures

<table>
<thead>
<tr>
<th>Particulars</th>
<th>40±0.5% total solid</th>
<th>50±0.5% total solid</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial Temperature of holding</td>
<td>Initial Temperature of holding</td>
</tr>
<tr>
<td></td>
<td>5°C</td>
<td>10°C</td>
</tr>
<tr>
<td></td>
<td>Period of holding (hhrs.)</td>
<td>Period of holding (hhrs.)</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>12</td>
</tr>
<tr>
<td>Dynamic viscosity (Cp)</td>
<td>29.25</td>
<td>34.94</td>
</tr>
<tr>
<td>Average No. of lactose crystals (millions/gm)</td>
<td>---</td>
<td>2.13</td>
</tr>
<tr>
<td>Average lactose crystal size (micron)</td>
<td>---</td>
<td>14.51</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSIONS

Results

1) Data obtained on viscosity and size and No. of lactose crystals in 50 per cent concentrate when stored at 50°C temperature for 12-14 hours is presented in Table VIII.

2) Data obtained on initial quality of skim milk and production details is presented in Table IX.

3) Data obtained on the effect of variation in speed of atomizer on the physical characteristics of instant milk powder (Buffaloes's milk powder) is presented in Table X.

4) Data obtained on effect of storage period on the physical characteristics of instant skimmed milk powder is presented in Table XI.

Discussion

1. Viscosity and size and no. of lactose crystals in buffaloes's skimmed milk concentrate

Data in Table 'VI. I' it is revealed that initial viscosity of 40 and 50 per cent total solids in concentrate was 29.25 and 43.30 cp. respectively. Initial viscosity of 50 per cent concentrate was 1.8 times more than that of the 40 per cent concentrate. When 40 per cent concentrate was stored at 50°C temperature for 10,12 and 14 hours the values of viscosity were 34.94, 39.0 and 45.0 cp respectively whereas as in the sample stored at 10°C the values corresponding to different storage periods were 32.60, 32.99 and 33.15 cp respectively.
The viscosity of samples stored for 10, 12 and 14 hours was increased by 18.4, 33 and 53 per cent respectively. The viscosity increased very nearly, proportionately to the increase in storage time.

In samples stored at 10° C, for 10, 12 and 14 hours the viscosity was found to be 32.50, 32.99 and 33.15 respectively. The increase in viscosity was observed to be 11.11, 12.78 and 13.33 per cent over the initial value. In case of 50 per cent concentrate, the initial viscosity was 43.20 cp when it was stored for 10, 12 and 14 hours at 5° C the viscosity rose to 77.71, 86.40 and 97.92 cp respectively. It is apparent from the above results that the viscosity increased by 80.00, 100 and 120.66 per cent over the initial viscosity.

In case of samples stored at 10° C for 10, 12 and 14 hours viscosity was observed to be 50.40, 63.30 and 64.00 cp respectively. The increase in viscosity over initial was found to be 16.66, 23.33 and 24.10 per cent corresponding to the storage periods.

It may be concluded from the above results that

a) The viscosity of 40 and 50 per cent concentrate increased with the storage periods at holding temperatures of 5 and 10° C.

b) The increase in viscosity was almost proportional to the increase in the holding periods at 5 and 10° C.
c) The viscosity of samples of 40 per cent concentrate stored at 5 and 10°C showed an increase by 53.00 per cent and 23.31 per cent over the initial viscosity of 29.25 cp during a storage period of 14 hours. In the case of 50 per cent concentrate, the increase in viscosity observed at 5 and 10°C was 125.0 and 54.10 of the initial value 43.20 cp during the storage period of 14 hours. The rate of increase of viscosity was more in samples stored at 5°C as compared to samples stored at 10°C.

4) Both the 40 and 50 per cent concentrates attained the maximum viscosity when stored for 14 hours.

As the temperature of concentrate drops, there is an inevitable tendency for the lactose to crystallize. This continues until a point is reached in the cooling process where the crystallizing effect of increasing supersaturation is offset, which in turn increases the viscosity and concentration of colloidal substance. The crystallization depends upon the amount of concentration of total solids, holding temperature and time. As the temperature drops lower, supersaturation increases and the rate of crystallization becomes more rapid.

**Number and size of lactose crystals in concentrate stored for varying periods**

Data in Table VIII shows that

1) Number of lactose crystals in 40 per cent concentrate stored at 5°C for 10, 12, and 14 hours was 2.13, 3.30 and 5.50 million per gram respectively, corresponding to
the period of storage. As such the number of crystals corresponding to the period at 10 hours of storage increased by 54.08 and 153.21 per cent over the number in the sample stored for 10 hours at 5°C.

2) The number of lactose crystals in 40 per cent concentrate sample stored for 10, 12 and 14 hours at 10°C was 1.14, 1.31 and 1.39 millions per gram respectively. The number of crystals corresponding to the 12th and 14th hours of storage increased by 12.38 and 19.00 per cent over the number in the sample stored for 10 hours at 10°C.

3) The number of lactose crystals in 50 per cent concentrate stored at 5°C for 10, 12 and 14 hours was 5.95 million, 8.93 million and 10.1 million respectively, corresponding to the period of storage. As such the number of crystals corresponding to the 12th and 14th hours of storage increased by 59.88 and 69.74 per cent over the number in the sample stored for 10 hours at 5°C. The number of lactose crystal in 50 per cent concentrate stored at 10°C for 10, 12 and 14 hours was 4.30 million, 5.50 million and 5.86 million per gram respectively. The number increased by 27.86 and 24.86 per cent over the number in samples stored for 10 hours at 10°C.

In general, samples stored at 5°C developed greater number of crystals than sample stored at 10°C corresponding to all the intervals of storage studies. Between concentration the growth appeared to be more in 50 per cent
concentrate than in 40 per cent concentrate. Between the periods of storage the maximum number of crystals were observed after maximum storage period of 14 hours at both the storage temperatures.

The above observations are in concurrence with the observations of Whittier (1944).

The average size of lactose crystals in 40 per cent concentrate stored at 5°C for 10, 12 and 14 hours was 14.51/μ, 15.60/μ and 16.50/μ respectively. The size of crystal in 40 per cent concentrate stored for 10, 12 and 14 hours at 10°C was 12.70/μ, 13.60/μ and 13.9/μ respectively. The size of crystals in sample stored at 8°C was slightly larger (by about 1.71/μ over to size of crystals in sample stored at 10°C).

The size of lactose crystals in 50 per cent concentrate stored at 5°C for 10, 12 and 14 hours was 23.10/μ, 25.08/μ and 25.83/μ respectively. The size of crystals in 50 per cent concentrate stored at 10°C was 19.61, 19.38 and 20.62/μ corresponding to the different storage periods.

The size of crystals in sample stored at 8°C was larger compared to the size of crystals in sample stored at 10°C. The rate of increase in crystal size of lactose was very slow at both the storage temperatures.

In general, it could be observed that a 50 per cent concentrate stored at 8°C for 14 hours gives the maximum number of large size crystals. The presence of large
<table>
<thead>
<tr>
<th>Quality of milk</th>
<th>Alcohol test</th>
<th>Acid test</th>
<th>Fat test</th>
<th>S.N.P.T.S.</th>
<th>Temperature</th>
<th>Time</th>
<th>'T. S.' Holding</th>
<th>Relevance data on drying</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>Speed of atomizer</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>in °C</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>hrs.</td>
</tr>
<tr>
<td>-ve</td>
<td>0.15</td>
<td>0.10</td>
<td>9.75</td>
<td>9.85</td>
<td>90</td>
<td>Flash</td>
<td>60</td>
<td>50</td>
</tr>
<tr>
<td></td>
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<td></td>
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<tr>
<td>-ve</td>
<td>0.14</td>
<td>0.10</td>
<td>9.74</td>
<td>9.84</td>
<td>90</td>
<td>Flash</td>
<td>60</td>
<td>50</td>
</tr>
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</tr>
<tr>
<td>-ve</td>
<td>0.16</td>
<td>0.05</td>
<td>9.72</td>
<td>9.77</td>
<td>90</td>
<td>Flash</td>
<td>60</td>
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</tbody>
</table>
Table X

Effect of variation in speed of atomization on the physical characteristics of instant non-fat buffalo's milk powder.

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Atomizer R.F.M. (± 1000)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>7000</td>
</tr>
<tr>
<td>1</td>
<td>Moisture %</td>
<td>3.95</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.032</td>
</tr>
<tr>
<td>2</td>
<td>Acidity %</td>
<td>0.11</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.012</td>
</tr>
<tr>
<td>3</td>
<td>Fat %</td>
<td>1.07</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.004</td>
</tr>
<tr>
<td>4</td>
<td>Solubility index (ml)</td>
<td>0.24</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.044</td>
</tr>
<tr>
<td>5</td>
<td>Particle size (micron)</td>
<td>72.4</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±1.140</td>
</tr>
<tr>
<td>6</td>
<td>Particle density (g/ml)</td>
<td>1.33</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.01</td>
</tr>
<tr>
<td>7</td>
<td>Bulk density (g/ml)</td>
<td>0.706</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.006</td>
</tr>
<tr>
<td>8</td>
<td>Wettability (time in sec.) per one gram powder</td>
<td>11</td>
</tr>
<tr>
<td>9</td>
<td>Sinkability (transmittance %)</td>
<td>2 mins. 56</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4 mins. 54</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 mins. 84</td>
</tr>
<tr>
<td>10</td>
<td>Dispersibility (g)</td>
<td>48.70</td>
</tr>
<tr>
<td></td>
<td>×××</td>
<td>±0.162</td>
</tr>
</tbody>
</table>

SE = Standard error
number of mass crystals of lactose is a factor which
determines the success of near instant character of
resulting powder in a single pass spray dried, therefore
the above represents the optimum requisites for pre-
condensing of the concentrate prior to drying.

**Instant Non-fat Buffalo's Milk Powder**

**II. Quality of Raw Milk**

Data in Table IV indicate that the quality of
raw milk used for the replicates in the production of
instant milk powder was almost uniform. Fat content
varied from 0.05 to 0.1 per cent.

**Production details (Fig. 2, flow diagram 2)**

Preheating temperature of milk was kept at 90°C.
The condensing was done at 80 ± 0.5 per cent total solids
and stored at 5°C for 12-14 hours in accordance with the
observation made from the laboratory experiment. Temp-
erature was kept at 200°C and cut 1st temperature was
95 ± 1°C. The speed of atomizer was varied as 7,000,
10,000 and 15,000 r.p.m.

**III. On the basis of results presented in
Table X, the following observations were made:**

a) **Moisture content**

It is apparent from the results (Fig.-23) that
the moisture percentage increased with a decrease in
number of revolutions per minute being 3.25 at 15,000
r.p.m., 3.61 at 10,000 r.p.m., and 3.95 at 7,000 r.p.m.
The relationship of moisture percentage and speed of atomization was found to be a negative correlation. The normal moisture content of a good quality non-fat instant powder as specified by A.C.M.I. is 5.0 per cent. Residence time remaining the same, the moisture content increased with decrease in speed of atomization.

b) Fat content

Fat content and acidity of the final powder are always dependent upon the initial quality of raw milk. From table X, it is seen that the fat and acidity contents were found to be within the limit of 1.25 per cent fat and 0.15 per cent titratable acidity required by A.C.M.I. standards for instant non-fat dry milk. (A product of standard quality with regards to fat and acidity could be obtained by using fresh milk of low initial fat content).

c) Solubility index

The results presented in Table X and figure 24 show that the solubility index values at 15,000, 10,000 and 7,000 r.p.m. were 0.7, 0.6 and 0.4 ml. respectively. According to the specification of A.C.M.I., the solubility index should not be more than 1.0 ml. for instant milk powder. It was found that the lower r.p.m. gave lower solubility index. Higher total solids concentration in pre-condensed milk gives bigger particles at 7,000 r.p.m. than at 15,000 and 10,000 r.p.m. resulting in higher moisture content at
low speed of atomization. The heat damage due to drying alone is less severe in high moisture powder particles than with smaller particles which have comparatively lower moisture content. Thus, solubility index being a function of heat damage, it was found to increase progressively with the increase in r.p.m. of atomization.

d) Particle size

The particle size of powder at atomization speed of 7,000, 10,000 and 15,000 r.p.m. was found to be 72.4, 54.1 and 42.8 μ respectively. When the other conditions in terms of initial viscosity, outlet temperature were kept constant, particle size is dependent on the speed of atomization. There was a negative correlation of speed of atomization with the size of powder particles. Average particle size above 40 micron and below 125 micron (Pyne, 1961) disperse most easily in water than the smaller or bigger particles. Perhaps an ideal product should contain particles of uniform size just large enough to permit reasonable rate of self-dispersion.

e) Particle density

The particle density of powder was found to be 1.33, 1.28 and 1.25 g/ml. corresponding to the speed of 7,000, 10,000 and 15,000 r.p.m. (Refer fig.25). This trend was in accordance with the fact that at lower speed of atomization results in bigger particles with higher moisture content and less entrapped air. An increase in bulk density is beneficial to reduce packing cost and for
EFFECT OF ATOMIZER SPEED ON PHYSICAL CHARACTERISTICS OF BUFFALO'S NON-FAT INSTANT MILK POWDER.

FIG. 23

REVOLUTION IN 1000 RPM

FIG. 24

REVOLUTION IN 1000 RPM

FIG. 25

REVOLUTION IN 1000 RPM

FIG. 26

REVOLUTION IN 1000 RPM

MOISTURE %

SOLUBILITY INDEX IN ml.

BULK DENSITY

DISPERSBILITY IN qm
the recovery of powder particles from the escaping air in cyclone collection. Density also contributes to the amount of particles that get air-born during drying, shifting and handling. Excessive dust in dry milk is the result of low particle density and it is, therefore, advisable to go for high bulk density.

f) Bulk density

Results indicate that the bulk density was 0.706 g/ml at 7,000; 0.673 g/ml at 10,000 and 0.650 g/ml at 15,000 r.p.m. It shows that the highest bulk density was obtained in powders of low speed atomization. Hanrahan and Konston (1965) reported that the bulk density of the agglomerated powder usually ranges from 0.26 to 0.37 g/ml. The present results for bulk density of the 'single-pass' nonfat dry milk indicated incomplete instancy obtained by the increase in the particles size, particle density and consequently the bulk density. Bulk density is one of the most important commercial criteria because it affects the size of standard containers, storage space and transport space. The single-pass drying technique which yields a product of considerably improved dispersibility at a relatively higher level of bulk density with bigger particle size appears to have greater practical value.
g) Wettability

The results presented in Table X and Fig. 26 show that the mean wetting time for 1 g of powder was observed to be 14 seconds at 7,000; 32 seconds at 10,000 and 64 seconds at 15,000 r.p.m. Various brands of instant non-fat dried milks have been reported to have shown a mean wetting time in range 68 to 10 seconds and few powders of larger particles and higher density gave times of 7 to 30 seconds as observed by Muers and House (1962). The powder made at 7,000 r.p.m. corresponds to the latter group. The 'single-pass' instant non-fat dry milk at low speed of atomization resulting in increased particle size by partial agglomeration is said to be a satisfactory instant product. Larger particles provide more space in the interstices for wetting. More uniformity of larger particles results in less time and consequently improved wetting. The crystallization of lactose before drying perhaps helps in improving the wettability by orienting itself to the surface along with the salts and provides the readily soluble components (Backian et al., 1957). The less soluble components, i.e. calcium-caseinate, are oriented towards the centre of the particles and are more readily dissolved after the salts and lactose have gone into solution. A study of Fig. 30 shows that the area remaining same, amount of powder wetting increased with log of time.
b) Sinkability

Results indicate that the maximum transmittance of light was attained within 2 minutes i.e. 56.61 and 66.8 per cent corresponding to the speed of atomizer. The low r.p.m. powder attained maximum opacity by this time and slight increase in opacity continued till 4 minutes. After six minutes when the cuvette was tapped six times on the sides a peculiar condition was found that the per cent transmittance increased rapidly to about 84 per cent in case of 7,000; 76 per cent in case of 10,000 and 71 per cent in case of powder from 15,000 r.p.m. of atomization. Since low speed of atomization is responsible for giving large particles, the individual particles probably keep further apart as they reach the surface of water. These heavier individual particles overcome the surface tension and sink rapidly into the water (Jambhayer, 1966). They are suspended in quiescent water but on tapping, sink to bottom quickly, resulting in the increased transmittance of light.

1) Dispersibility

The dispersibility values of 48.70 g, 46.77 g and 45.37 g have been obtained taking 52 g sample from milk powder produced at atomization speed of 7,000; 10,000 and 15,000 r.p.m. respectively, which represent about 92.6, 90.0 and 87.5 per cent respectively for the variable speeds of atomization. As against the requirement of 64 g. by
1.0 M.I. using low energy method, the data obtained on
dispersibility in present study was higher because of the
fact that the mixture employed for the determination had
a r.p.m. of 400 against the 1.0 M.I. specification of 192
r.p.m. The higher values obtained substantiate Howiet's
(1963) claim that speed of stirring is more important
than the duration of stirring used to disperse milk
solids. High rate of stirring maintains the concentra-
tion of milk solids around the dissolving powder at levels
which do not cause destabilization of milk protein system.

The drop size depends upon the velocity (decreas-
ing with increasing velocity) of milk during atomization
by altering the speed of the atomizer. In general, however,
centrifugal atomizers tend to produce powders of larger
average particle size than that of jet types; the range
is from 50 to 250μ (Coulter, 1964). The bulk density of
the powder particles is increased whilst the volume of
entrapped air is reduced as more concentrated milk at low
speed is sprayed by the centrifugal atomizer. Moreover,
the powder manufactured in this condition tends to produce
a high proportion of large particles which help to diss-
olve in water more easily. It has also been claimed that
a greater degree of uniformity of particle size is
obtained which in turn improves the wettability, sinkabi-
liy and dispersibility.
Normal versus Instant Buffalo Milk Powder

A comparison of the data obtained on physical characteristics of the normal and instant non-fat dry milk leads to the following conclusions:

1) Moisture content of normal non-fat dry milk was 9.3 per cent in highest concentrate i.e. 50 per cent total solids. A 50 per cent total solids used for making instant milk powder gave maximum moisture content of 3.95 per cent at the lowest level of atomizer speed i.e. 7,000 r.p.m.

2) Operating conditions remaining the same, solubility index increased with increase in maximum value of 0.87 ml at 50 per cent of total solids in concentrate. At 50 per cent total solids concentrate in instant non-fat dry milk, solubility index became a function of atomization speed showing 0.4 ml at 7,000 r.p.m. and 0.7 ml at 15,000 r.p.m.

3) Biggest particle size of 33.33 micron obtained in normal spray dried powder at 50 per cent total solids in the concentrate was almost double that of instant milk powder (72.4 micron) made at an atomization speed of 7,000 r.p.m. using the same level of concentration in precondensed milk. This, therefore, was in favour of instant milk powder, as it was based on the improvement of physical characteristics through increase in size.

4) Improvement in particle density of normal milk powder resulted when a high total solids concentrate gave a value of 1.08 g/ml at 50 per cent total solids level.
Using the same per cent total solids on concentrate for instant milk powder production, a further improvement was affected when it attained a value of 1.33 g/ml at 7,000 r.p.m. of atomization speed which progressively decreased as the speed increased. Bulk density also exhibited a trend for higher values at higher level of preconcentration of milk. A value of 0.45 g/ml obtained for normal non-fat dry milk at 50 per cent total solids in the precondensed milk showed a further increase to 0.706 g/ml at 7,000 r.p.m. of atomization in instant non-fat dry milk made from milk precondensed to the same level.

5) The 'balling up' tendency of normal milk powder was observed to be absent in instant milk powder and it took less time to wet a given amount of latter type than its normal counterpart. The empirical wetting times obtained served as clear demarcating lines between instant and non instant type of milk powders produced. From the wettability test employed, it was clear that most important factor in determining the case of wetting was the size of the powder particles. A perferrential orientation of the crystalized lactose towards the outer surface is thought to be responsible for improving the wettability of instant powder as against the amorphous condition of lactose present in the normal milk powder.

6) The per cent transmittance values for normal and instant non-fat dry milks showed that instant dry milk attained
maximum opacity in 2-4 minutes against the continuous increase of opacity with time in case of normal milk powder. Increase after 6 minutes shows that mechanical agitation was necessary to put the instant skim milk powders bigger particles in solution.

7) Dispersibility values obtained for normal milk powder revealed a progressive increase with rise in per cent total solids in the precondensed milk, giving 35.92 g. at 50 per cent total solids level. A further increase has been observed when the 50 per cent total solid concentrate was atomized at 7,000 r.p.m. after allowing lactose crystallization by holding it over 14-16 hours at 5°C for instant milk powder production. The latter has been found to yield particles of almost double the size of former. Since dispersion time increased with increase in the size we should anticipate a lower value. The high observed value was perhaps due to the fact that a high speed agitation has been employed and the speed of agitation is more important that the time of agitation (Stone et al., 1954).

IV. Effect of storage period on the physical properties of instant skimmed milk powder
a) Moisture
The results in Table XI show that the moisture content of powder increased steadily from 3.95 per cent to 4.35 per cent at the end of 24 weeks of storage. The overall increase in moisture per cent during storage was found to be 9.2 per cent. The reason for slow increase in moisture was the obvious absorption of moisture through the walls of
Table XI

Effect of storage on physical properties of Instant skimmed milk powder (Temp. 28° - 32°C)

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>0</th>
<th>4</th>
<th>8</th>
<th>12</th>
<th>16</th>
<th>24</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Moisture (%)</td>
<td>3.95</td>
<td>3.82</td>
<td>3.98</td>
<td>4.26</td>
<td>4.29</td>
<td>4.35</td>
</tr>
<tr>
<td>2.</td>
<td>Acidity (%)</td>
<td>0.11</td>
<td>0.11</td>
<td>0.11</td>
<td>0.12</td>
<td>0.13</td>
<td>0.14</td>
</tr>
<tr>
<td>3.</td>
<td>Solubility index (ml)</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.5</td>
<td>0.6</td>
<td>0.8</td>
</tr>
<tr>
<td>4.</td>
<td>Wettability (seconds)</td>
<td>14</td>
<td>14</td>
<td>14</td>
<td>14</td>
<td>14</td>
<td>14</td>
</tr>
<tr>
<td>5.</td>
<td>Sinkability (transmittance %)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2 mins.</td>
<td>56</td>
<td>56</td>
<td>58</td>
<td>59</td>
<td>61</td>
<td>63</td>
</tr>
<tr>
<td></td>
<td>4 mins.</td>
<td>52</td>
<td>54</td>
<td>53</td>
<td>58</td>
<td>59</td>
<td>61</td>
</tr>
<tr>
<td></td>
<td>6 mins.</td>
<td>84</td>
<td>83</td>
<td>81</td>
<td>80</td>
<td>80</td>
<td>78</td>
</tr>
<tr>
<td>6.</td>
<td>Dispersibility (gm)</td>
<td>48.65</td>
<td>48.70</td>
<td>48.50</td>
<td>48.30</td>
<td>47.20</td>
<td>47.80</td>
</tr>
</tbody>
</table>
the pack. However, when the packing was in polycraft bags with inner lining of polythene, the absorption of moisture was quite within permissible limits and the powder had a moisture level much below the prescribed minimum of 5 per cent (A.D.M.I).

b) Acidity

The results in Table XI on acidity of reconstituted milk of instant milk powder, indicate that the acidity had increased from .11 to .14 i.e. by 27.27 per cent. The increase was very slow during the storage period. This increase in acidity may be due to the interaction between lactose and free-amine groups of the protein. This reaction is helped by moisture content and high storage temperature (Kathleen, 1949).

c) Solubility index

It is apparent from Table XI and Fig. that the solubility index increased from 0.4 ml to 0.8 ml i.e. by only 0.4 ml, which is not very appreciable. Similar results have been reported by Abbot and Waite (1962) for Cow’s skimmed milk instant milk powder.

4) Wettability

The results obtained on wettability show that the wettability (i.e. time to wet 4 gram powder) was not affected by storage of skimmed milk powder.

e) Sinkability

The results indicate that the sinkability of powder at the end of storage period at 2 minutes was found 63 per cent transmittance and at 6 minutes it was 78 per cent.
while initial values corresponding to 2 minute was 56 per cent and at 6 minute 83 per cent transmittance. There appears to be a decrease in transmittance by 7 per cent at 2 minutes and 5 per cent at 6 minutes.

f) Dispersibility

The dispersibility was found to decrease from 43.70 to 47.8 g., the decrease in dispersibility being only by 1.30 per cent.

The results, in general, show that the characteristics of instant powdered do not undergo any considerable change during storage for 24 weeks at 23-30°C. Abbott and Waite, while studying the physico-chemical changes in instant skim milk powder have recorded similar findings. The most important factor which affects the dispersibility during storage is the moisture content of the powder. Lea and White (1943) found that the solubility of dried skim milk with moisture content of 3.0 per cent and 5.0 per cent remained almost same or unaltered after 700 days at 37°C.
Studies on physical characteristics of roller and spray dried whole milk powder as influenced by conditions of manufacture and storage (cow and buffalo)
SECTION IV

ROLLER DRIED AND SPRAY DRIED WHOLE MILK POWDER

Raw whole milk was collected from contractors who collect the milk from farmers within 20 miles radius of Karnal. The fat per cent of buffalo's milk was 7 to 7.5 per cent. The cow's milk was supplied by Dairy Husbandry Division, National Dairy Research Institute, Karnal to Experimental Dairy, Dairy Technology Division with 5 to 5.5 per cent fat.

EXPERIMENTAL

a) Selection of whole milk powder

Procedure followed for selection of whole milk was the same as described in Section II.

b) Standardization of milk

It was desired that the products should conform to the I.S.I. specification (I.S. 1165-1957).

For the manufacture of whole milk powder, it was aimed to standardize raw milk to have 3.5 per cent fat. It was achieved by separating a part of the fat from whole milk and blending it in proper proportion after testing to give 3.5 per cent fat. Quantities of whole milk and skim milk to be taken were mixed and kept agitated in Decco Cooler, until preheating.

c) Preheating

Temperature of preheating was kept at 90°, 95° and 115° C in flash type of plate heater.
d) Condensing

Equipment and technique of condensing were the same as in earlier studies except that the total solids in the final concentrate was kept to the 30 ± 0.5 per cent.

e) Homogenization

Concentrated milk was homogenized at 160 Kg pressure per square cm in first stage and in second stage, the pressure was 200 Kg pressure per square cm.

f) Spray-drying

The procedure to manufacture whole milk powder was followed as in case of skimmed milk powder.

A. Sampling

As per the plan of the experiment 12 tins of each samples and trial were collected in tins, each tin containing 400 grams of powder. These tins were sealed immediately.

All the sealed sample containers were put inverted on the tray of the gas chamber, and a hole of about 2 cm diameter was punched in the bottom of each tin. The tins were subjected to the treatments as follows:

The tray containing the inverted punctured tins was pushed inside the vacuum chamber provided by Anhydros Company along with Anhydros Spray Dried Plant for gas packing. After closing the chamber, the vacuum pump was started. It was held at maximum attainable (720 mm in this case) vacuum for 1 minute. The vacuum pump was stopped and the nitrogen
gas (99.9 per cent purity) was let into the chamber at a pressure of 100 mm over the atmospheric pressure, for one minute.

The tray was taken out and the tins soldered to quickly close the holes in the bottom. The same process was repeated after 24 hours for double gas packing in order to reduce the oxygen content in head space of the tins to about 2 to 3 per cent.

Storage

All the tins (air and gas packed) were stored at 30°C and 32°C for 12 months.

Number of trials

Three trials of each treatment were made.

B. Analytical

Preconcentrated milk

Initially during the condensing operation, the total solids were determined with the help of 'Bausch and Lomb' refractometer by putting a drop of well mixed sample on the clean prism, closing it and directly reading the total solid contents on the scale.

The total solids content was later confirmed by the gravimetric determination on Mojonier tester as detailed for unsweetened condensed milk by Milk Industry Foundation in Methods of Analysis of Milk and its Products (Laboratory Manual) 3rd Edition.

Dried Milk

Dried whole milk (Roller/Spray) was tested immediately after manufacture and intervals of 2 months in storage studies.
I) **Smell**

Immediately as the container was opened, it was tested for smell and flavour.

II) **Colour**

A modified method of Doob (1942) was used for evaluation of the intensity of colour of milk powder as described in Section I.

III) **Moisture content**

The gravimetric procedure on Mojonnier tester was followed as described in Laboratory Manual, 3rd Edition.

IV) **Fat content**

Fat content was determined only on the fresh product for confirmation. Procedure was followed as described in Laboratory Manual 3rd Edition.

V) **Free fat**

Free fat in powder was determined by adding 100 ml petroleum ether to 10 gm of sample in 250 ml. Erlenmeyer flask. It was shook ten times. Then it was allowed to settle for 15 minutes. The petroleum ether layer was filtered through No. 42 Whatman filter paper catching the solvent in a tared Mojonnier fat dish. Second extraction was obtained by the same procedure. Ether was evaporated, the sample was weighed and result was calculated as percentage of free fat.
vi) Bulk density

A method of Sjollema (1963) to measure bulk density of powder was used. The method has been described in Section I.

vii) Solubility index

Solubility index of roller and spray dried whole milk powder was determined by the A.D.M.I. Method. Thirteen grams of the whole milk powder was taken to determine the solubility index which measures the volume sediment obtained on centrifugation of a specific volume of reconstituted milk. The method of centrifugation was same as used in determining the solubility index of skimmed milk powder (Section I).

viii) Wettability

The wettability of whole milk powder, which depends upon sinkability, was determined by a method suggested by Samhammer (1966). Distilled water 3.5 ml at 20°C was taken in cuvettes on the surface to which was dusted 13 mg of whole milk powder and percentage of transmittance was measured at 760 milli-micron wavelength in a Beckman D.V. Spectrophotometer after 2 and 4 minutes and then after 6 minutes after giving 6 tappings on the side.

ix) Dispersibility

The A.D.M.I. Method was adopted to determine the dispersibility of powder as described in Section I.
x) Analysis of Oxygen content in head space of stored can

Determination of oxygen present in head space of hermetically sealed, gas packed containers of whole milk powder was done by modified Orsat gas analyzer (A.D. M.I. Method, 1965).

xi) Peroxide value:

The ferric thiocyanate method of Hill and Thiel (1946) was followed for the peroxide value determination and it has been expressed as milliequivalents of oxygen per kg. of the powder.
<table>
<thead>
<tr>
<th>Particulars</th>
<th>Quality of raw milk fat (%)</th>
<th>Reheating fat (%)</th>
<th>T.S. (%) in concentrate</th>
<th>Temp. (°C)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cow SE</td>
<td>3.58</td>
<td>12.73</td>
<td>27</td>
<td>80</td>
<td>115</td>
</tr>
<tr>
<td>SE</td>
<td>3.06</td>
<td>12.27</td>
<td>50</td>
<td>80</td>
<td>115</td>
</tr>
<tr>
<td>Buffalo SE</td>
<td>3.63</td>
<td>12.39</td>
<td>27</td>
<td>80</td>
<td>95</td>
</tr>
<tr>
<td>SE</td>
<td>3.05</td>
<td>12.27</td>
<td>50</td>
<td>80</td>
<td>115</td>
</tr>
</tbody>
</table>

**Conditions pertaining to steam pressures in rollers and inlet air and outlet air temperatures in spray drier kept same as they were in manufacturing of skimmed milk powder.**

SE = Standard error
RESULTS AND DISCUSSION

Results

i) Data on quality of standardized whole milk powder are presented in Table XIII.

ii) Data on effect of different preheating and drying conditions on the physical characteristics of roller and spray dried whole milk powder are presented in Table XII and XIV.

iii) Data on effect of storage temperatures on the rate of oxygen absorption and peroxide developments in whole milk powder under different conditions of packing and storage are presented in graphs 31 to 33.

iv) Effect of storage temperatures on physical characteristics of whole milk powder (roller/spray).

Discussion

1. Quality of Raw Whole Milk Powder

Table XII show that the quality of raw milk (standardized milk) for the replicates in the production of roller dried whole milk powder was almost uniform. Fat content was varied from 3.46 to 3.53 per cent (recommended fat content is 3.50 per cent in milk).

Production details

From Table XII indicate that preheating temperatures were kept constant during preheating the milk i.e. 80°, 95° and 116° C.
Table VIII

Effect of different pre-heating and drying conditions on the physical characteristics of roller dried whole milk powder
(Cow and Buffalo)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>80°C Steam pressures</td>
<td>95°C Steam pressures</td>
<td>80°C Steam pressures</td>
<td>95°C Steam pressures</td>
</tr>
<tr>
<td></td>
<td>(in PSI)</td>
<td>(in PSI)</td>
<td>(in PSI)</td>
<td>(in PSI)</td>
</tr>
<tr>
<td>Colour</td>
<td>3+</td>
<td>3+</td>
<td>3++</td>
<td>3++</td>
</tr>
<tr>
<td>Flavour</td>
<td>Normal</td>
<td>Normal</td>
<td>Normal</td>
<td>Normal</td>
</tr>
<tr>
<td></td>
<td>SC</td>
<td>SC</td>
<td>SC</td>
<td>SC</td>
</tr>
<tr>
<td>pH</td>
<td>6.70</td>
<td>6.70</td>
<td>6.70</td>
<td>6.65</td>
</tr>
<tr>
<td></td>
<td>6.70</td>
<td>6.70</td>
<td>6.70</td>
<td>6.65</td>
</tr>
<tr>
<td>Fat (%)</td>
<td>27.68</td>
<td>27.73</td>
<td>27.92</td>
<td>27.81</td>
</tr>
<tr>
<td></td>
<td>±0.18</td>
<td>±0.13</td>
<td>±0.21</td>
<td>±0.18</td>
</tr>
<tr>
<td>Moisture (%)</td>
<td>3.32</td>
<td>3.12</td>
<td>3.23</td>
<td>3.18</td>
</tr>
<tr>
<td></td>
<td>±0.23</td>
<td>±0.16</td>
<td>±0.21</td>
<td>±0.25</td>
</tr>
<tr>
<td>Bulk density (g/ml)</td>
<td>0.301</td>
<td>0.302</td>
<td>0.303</td>
<td>0.304</td>
</tr>
<tr>
<td></td>
<td>±0.021</td>
<td>±0.026</td>
<td>±0.016</td>
<td>±0.011</td>
</tr>
<tr>
<td>Solubility index (ml)</td>
<td>12.5</td>
<td>13.2</td>
<td>12.7</td>
<td>13.2</td>
</tr>
<tr>
<td></td>
<td>±0.13</td>
<td>±0.21</td>
<td>±0.20</td>
<td>±0.18</td>
</tr>
<tr>
<td>Free fat (%)</td>
<td>83.23</td>
<td>83.16</td>
<td>86.81</td>
<td>90.31</td>
</tr>
<tr>
<td></td>
<td>±3.25</td>
<td>±1.25</td>
<td>±5.26</td>
<td>±1.27</td>
</tr>
</tbody>
</table>

SC = Slightly cooked; SE = Standard error
The steam pressure in rollers i.e. 60 and 80 p.s.i. were kept constant in case of drying of skimmed milk on rollers. In case of spray dried, inlet air temperatures were 120°C and 200°C and outlet temperature was kept 95°C.

II. Effect of different pre-heating and drying conditions on the physical characteristics of roller-dried whole milk powder

a) Colour

Observation on colour recorded in Table XII show that the roller-dried whole milk powder manufactured from cow’s whole milk pre-heated at 80°C and dried at 60 and 80 p.s.i. gave colour corresponding to the colour standard of 3++. The same milk pre-heated at 95°C and converted into powder using 60 and 90 p.s.i. gave colour corresponding to the standard 3+++.

Buffalo’s whole milk powder manufactured from milk preheated to 80°C and dried at 60 and 80 p.s.i. gave colour corresponding to the standard of 2++. While the same milk sample preheated at 95°C and dried at 60 and 80 p.s.i. gave corresponding to the standard 2+++.

The trend of changes found in whole milk powder were the same as in skimmed milk powder (discussed in Section I). However, the colour of powder from cow’s whole milk was more yellow than that of powder from cow’s skimmed milk because of carotene associated with the fat in cow’s whole milk.

b) Moisture

The data on moisture content in Table XIII indicate that the moisture content of whole milk powder from cow’s
milk preheated at 80°C and dried at 60 and 80 p.s.i. was found to be 3.32 per cent and 3.12 per cent corresponding to their low roller pressures. Similar milk preheated at 95°C and dried at 60 and 80 p.s.i. gave moisture content of 3.23 per cent and 3.18 per cent respectively.

Whole milk powder from buffalo milk preheated at 80°C and dried at 60 and 80 p.s.i. gave moisture content of 3.41 per cent and 3.16 per cent corresponding to the low steam pressures used. When the same milk was preheated to 95°C and dried at 60 and 80 p.s.i., the moisture content was found to be 3.33 per cent and 3.08 per cent respectively.

The trend of variation in moisture content of fresh whole milk powder was found to be same as in case of corresponding skimmed milk powder.

c) Bulk Density

The data on bulk density in Table 4.11 show that the bulk density of whole milk powder from cow's milk subjected to preheating of 80°C and steam pressure of 60 and 80 p.s.i. were 0.301 g/ml and 0.302 g/ml. Same milk when preheated to 95°C and dried at 60 and 80 p.s.i. gave bulk density 0.303 g/ml and 0.301 g/ml corresponding to the steam pressure.

In milk powder from buffalo's milk preheated to 80°C and dried at 60 and 80 p.s.i., the bulk densities were found to be 0.312 and 0.305 g/ml corresponding to the two roller pressures. When the same milk was subjected to 95°C preheating temperature and 60 and 80 p.s.i. steam pressure, the values for bulk density were 0.304 and 0.313 g/ml respectively.
The above observations show that the values for bulk density of either type of milk overlapped and there was no appreciable affect of pre-heating temperature and steam pressure on bulk density. However, the bulk density of whole milk powder of either type of milk was less by 0.02 to 0.03 g/ml as compared to the corresponding roller dried skimmed milk powder. This difference could be attributed to the fat percent in the whole milk powder. The discussion relating to the influence of other factors like grinding on bulk density of roller dried skimmed milk (refer Section 1) apply equally to roller dried powder from whole milk.

4) Solubility Index

It is apparent from data in Table XII that roller dried whole milk powder from cow's milk preheated at 80°C and dried at 60 and 80 p.s.i. pressure, the solubility index was found 12.5 ml and 13.2 ml corresponding to the two steam pressures used for drying. Same milk when subjected to 95°C pre-heating temperature and 60 and 80 p.s.i. roller pressures, the solubility index were found to be 12.7 ml and 13.4 ml respectively.

Whole milk powder manufactured from buffalo's milk preheated at 80°C and dried at steam pressures of 60 and 80 p.s.i. gave solubility index 13.3 ml and 13.8 ml corresponding to these two steam pressures. When the same milk was preheated to 95°C, the values for solubility index were 13.4 and 14.0 ml.
The trend of results was found to be same as in the case of roller dried skimmed milk powder (Section 1). High preheating temperature was found to increase the solubility index very slightly. This slight increase in solubility index resulting in consequent lowering of dispersibility should not be of much consequence since a higher preheating temperature prolongs the keeping quality of whole milk powder (Findlay et al, 1946).

Free Fat

It is seen from Table XIII that free fat was from 83.23 to 91.23 per cent in cow’s milk powder, while in case of buffalo’s milk powder was from 84.8 to 89.27 per cent. The results indicate that the high steam pressure gave more free fat as compared to low steam pressure in either of milk powders. It shows that the most of fat was free and much of it was on the surface of the particles. This high percentage may be caused by rupturing the globule membrane by the hot drum and scraping action of knife in removing the film. The free fat is also increased by increasing the fat content of milk, exposure to high steam pressure and to the pulverizing in hammer mill and holding dry product in a hot condition. Choi et al (1951) suggested that coagulation of the protein membrane contributed to freeing the fat.

Effect of different preheating and drying conditions on the physical characteristics of freshly dried (spray) whole milk powder.

a) Colour

The observation on colour of powder in Table XIV show that milk preheated at 90°C gave the colour 3+ in cow’s
### Table XII

Effect of different pre-heating and drying conditions on the physical characteristics of spray dried whole milk powder (Cow and Buffalo).

<table>
<thead>
<tr>
<th>Particulars</th>
<th>Cow 80°C</th>
<th>Cow 115°C</th>
<th>Buffalo 95°C</th>
<th>Buffalo 115°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>180°C</td>
<td>200°C</td>
<td>180°C</td>
<td>200°C</td>
</tr>
<tr>
<td>Colour</td>
<td>3*</td>
<td>3*</td>
<td>3*</td>
<td>3*</td>
</tr>
<tr>
<td>Flavour</td>
<td>Normal</td>
<td>Normal</td>
<td>SC</td>
<td>SC</td>
</tr>
<tr>
<td>Fat (%)</td>
<td>27.87 ± 0.16</td>
<td>27.78 ± 0.19</td>
<td>27.91 ± 0.13</td>
<td>27.89 ± 0.15</td>
</tr>
<tr>
<td>Moisture (%)</td>
<td>1.53 ± 0.21</td>
<td>0.92 ± 0.13</td>
<td>1.43 ± 0.18</td>
<td>0.83 ± 0.31</td>
</tr>
<tr>
<td>Bulk density (g/ml)</td>
<td>0.438 ± 0.027</td>
<td>0.431 ± 0.021</td>
<td>0.438 ± 0.018</td>
<td>0.436 ± 0.025</td>
</tr>
<tr>
<td>Solubility index (ml)</td>
<td>0.20 ± 0.00</td>
<td>0.50 ± 0.10</td>
<td>0.40 ± 0.10</td>
<td>0.80 ± 0.20</td>
</tr>
<tr>
<td>Wettability (2 min)</td>
<td>75</td>
<td>79</td>
<td>76</td>
<td>78</td>
</tr>
<tr>
<td>(in transmittance %)</td>
<td>70</td>
<td>71</td>
<td>73</td>
<td>72</td>
</tr>
<tr>
<td>Dispersibility (g)</td>
<td>26.32 ± 2.38</td>
<td>27.51 ± 2.28</td>
<td>27.83 ± 2.58</td>
<td>27.16 ± 2.83</td>
</tr>
<tr>
<td>Free fat (%)</td>
<td>8.53 ± 1.36</td>
<td>9.68 ± 1.81</td>
<td>8.62 ± 1.31</td>
<td>9.88 ± 1.33</td>
</tr>
</tbody>
</table>

SC = Slightly cooked; SE = Standard error

Outlet temperature was kept constant at 95±10°C in all trials.
whole milk powder and 2 in case of buffalo's whole milk powder irrespective of inlet air temperature i.e. 180°C and 200°C.

Same milk, preheated at 95°C gave the 3++ in cow's whole milk powder and 2+ in buffalo's whole milk powder having kept same inlet air temperature.

When milk was preheated at 115°C, the colour of cow's whole milk powder was found 3+++ and that of buffalo's whole milk powder was 2++ irrespective of inlet air temperatures i.e. 190 and 200°C.

b) Flavour

It is apparent from data on flavour in Table XIV that the high preheating temperatures of 95°C and 115°C gave cooked flavour in either type of whole milk powders. However, the preheating temperature of 115°C gave a greater intensity of cooked flavour as compared to 95°C preheating with either type of milk. The flavour of powder manufactured from milk preheated at 90°C was found to be normal in either type of milk.

Sulphhydryl compounds are liberated from serum proteins probably from β-lactoglobulin. The volatile sulphides are associated with cooked flavour. Gould and Sommer (1939) established that at high preheating temperatures, the liberation of sulphhydryl compounds are prolonging the shelf life of whole milk powder (Finland and Mattick, 1948).
e) Moisture

The data on moisture in Table XIV show that the moisture content of powder manufactured from cow's milk preheated at 80, 95 and 115°C and dried at 130°C inlet temperature was found to be 1.53 per cent, 1.43 per cent and 1.47 per cent, respectively. When same milk was preheated to the same set of temperatures and dried at 200°C inlet temperature, the moisture content was 0.92 per cent, 0.83 per cent and 0.96 per cent corresponding to the three preheating temperatures respectively, it shows that low inlet air temperature i.e. 130°C gave about 35.61 to 39.80 per cent more moisture in powder. In the case of powder manufactured from buffalo's milk preheated at 80, 95 and 115°C and dried at 130°C inlet temperature, the moisture contents was 1.32, 1.25 and 1.35 per cent, respectively. When same milk was preheated at same set of preheating temperatures and dried at 200°C inlet air temperature, the moisture content of powder was 0.85, 0.93 and 0.88 per cent corresponding to three preheating temperatures respectively. It appears that increase in the temperature of inlet air by 20°C results in the reduction of final moistures in powder by 25.56 per cent to 36.30 per cent. The trend of results of moisture were same as found in case of spray dried skimmed milk powder.

d) Particle size

It is observed from Table XIV that i) the preheating temperature had no affect on particle size of either type of milk ii) at 200°C inlet air temperature, the particle
size of powder was found less as compared to powder (dried at 130°C inlet air temperature). The reason for this has been discussed in Section II, III. The particle size of all the samples of either type of milk was below 40 microns.

Ashworth (1965) reported that particle size of powder is most important factor influencing the wettability. 50 to 75 micron size is desired for good wettability.

e) Bulk density

The data in Table A IV indicate that the bulk density of powder manufactured from cow's whole milk preheated at 80, 95 and 115°C and dried at 130°C (inlet air temperature), was found to be 0.438 g/ml, 0.438 g/ml and 0.442 g/ml, respectively. The same milk preheated over same set of preheating temperatures and dried at inlet air temperature of 200°C had bulk densities of 0.431 g/ml, 0.436 g/ml and 0.432 g/ml corresponding to the three preheating temperatures respectively i.e. overall decrease in bulk density 0.02 to 0.1 g/ml at 200°C inlet air temperature.

The bulk density of powder prepared from buffalo's whole milk preheated at 80, 95 and 115°C and dried at 130°C inlet air temperature was 0.443 g/ml, 0.441 g/ml and 0.442 g/ml respectively. Same milk when preheated to similar preheating temperatures and dried at 200°C (inlet air temperature) gave bulk density of 0.438 g/ml, 0.439 g/ml and 0.438 g/ml corresponding to the three preheating temperatures respectively.
It may, therefore, be concluded from the above observations that the bulk densities of cow’s and buffalo’s milk powder overlap and temperatures of preheating have no appreciable influence on the bulk densities. However, at high inlet temperature, the bulk density of powder was found to be low as compared to powder dried at the low inlet temperature, the reason for this has been discussed in Section III. Bulk density of whole milk powder from either type of milk was found to be low by 0.20 to 0.30 g/ml as compared to corresponding samples of skimmed milk powder. This difference is mostly the result of the fat present in whole milk powder.

f) Solubility index

The observations on solubility index in Table X-V indicate that the solubility index of whole milk powder manufactured from cow’s milk preheated at 80, 90, and 115°C and dried at 150°C (inlet air temperatures) was 0.20 ml, 0.40 ml and 0.80 ml respectively. When the same sample of milk was preheated over the same set of preheating temperatures and dried at 200°C (inlet temperature) gave solubility index values of 0.50 ml, 0.80 ml and 1.0 ml. It may be concluded from the above results that when other conditions such as type of milk, atomizer speed, outlet air temperature etc. remain the same the solubility index is influenced more by the inlet air temperature than by the temperatures used for preheating of milk (range 80-115°C). However, between
samples of powder prepared by using different preheating temperatures there is a slight increase in solubility index with adoption of higher preheating temperatures.

In case of powder manufactured from buffalo's whole milk preheated at 80, 95 and 115°C and dried at 180°C (inlet air temperature), the solubility indices were 0.60 ml, 0.70 ml and 1.0 ml respectively. The solubility index of powder from milk preheated over same set of preheating temperatures and dried at 200°C were found to be 0.9 ml, 1.2 ml and 1.6 ml corresponding to the three temperatures of preheating.

Between powders made from two different types of milk, the product from buffalo's milk registered an increase in values of solubility index by 150-200 per cent over the values obtained for corresponding samples of cow's milk. Reasons for such differences in the solubility index of cow's and buffalo's milk have been discussed in the earlier section 4.

Finland and Mattick (1945) have also observed similar effects of preheating and inlet air temperatures in their work on spray dried cow's milk.

g) Dispersibility

The data on dispersibility of whole milk powder in Table XIV show that the values for powder from cow's milk preheated at 80°C, 95°C and 115°C and dried at 180°C (inlet air temperature) were 23.32 g, 27.33 g, and 27.64 g. Other conditions remaining the same when the temperature of
inlet air was increased to 200°C the values were 27.51 g, 27.16 g and 26.31 g corresponding to the three different types of pre-heating.

In case of powder manufactured from buffalo's milk preheated to 80, 95 and 115°C, the values for dispersibility were 25.33 g, 25.53 g and 25.16 g respectively when the temperature of inlet air was 180°C. When the same milk was preheated over same set of temperatures and dried at 200°C (Inlet temperature) the values for dispersibility were 25.86 g, 24.81 g and 24.43 g corresponding to the three types of preheating.

It may, therefore, be concluded that the dispersibility of powder from cow's milk was higher by 1.8 to 2.2 g as compared to similar samples from buffalo's milk. The discussion in Section II on the effect of particle size of spray dried powder from cow's and buffalo's milk on their dispersibility apply in general to the observations recorded in this section on the particles size of whole milk powder from either type of milk.

Hori and Hedrick (1965) reported that dispersibility of cow's whole milk powder (36 per cent fat) is the lowest (31.9 g) compared to products with lower fat contents.

h) Wettability

It appears from Table XIV that cow whole milk powder prepared from milk preheated at 80, 95 and 115°C and dried at 180°C inlet air temperature showed wettability in transmittance at 2 minutes, 75, 75 and 76 per cent and at
6 minutes the transmittance percents were 60.69 and 58 respectively whereas the powder prepared from milk at 200°C the transmittance percents were 79.78 and 80 at 2 minutes and at 6 minutes, the values were 71.72 and 72 per cent corresponding to the three pre-heating temperatures.

In case of buffalo's whole milk powder prepared from milk preheated at 90, 95 and 115°C and dried at 180°C, the values of wettability were found to be 78.79 and 81 at 2 minutes and at 6 minutes, the values were 72.71 and 72 per cent corresponding to the three preheating temperatures. Whereas the powder preheated at same set of preheating temperatures, the wettability was 81.85 and 85 per cent at 2 minutes and at 6 minutes, the values were 74.74 and 75 per cent corresponding to the preheating temperatures. The above results show that wettability of buffalo's milk powder was less as compared to cow's milk powder, because buffalo milk powder had more percentage of free fat. As inlet temperature and preheating temperatures increased, the wettability was found to be decreased. Mol et al (1962) and Sam Hammer also found that as free fat increased in powder, the wettability was decreased.

1) **Free fat**

The data from the present investigation on free fat (Table XV) show that the values for free fat in powder manufactured from cow's milk preheated to 90, 95 and 115°C and dried at 180°C (inlet temperature) were 8.53 per cent,
8.62 per cent and 8.39 per cent respectively. When the temperature of inlet air in drier was increased to 200°C, maintaining the other conditions same as above, the values for free fat changed to 9.69, 9.88 and 10.23 per cent corresponding to the preheating temperature studied.

Powder from buffalo milk preheated to 80, 95 and 115°C and dried at 120°C (inlet temperature) gave the values of free fat 9.23, 9.51 and 9.72 per cent corresponding to the different temperatures of preheating. A rise of 20°C in inlet air temperature, maintaining the other conditions of preheating same as above, increased the free fat in resulting powder to 9.84, 10.28 and 10.39 per cent corresponding to the different temperatures of preheating of milk.

It is evident from the above results that (i) as temperature of preheating increased, the values of free fat increased through slightly with either type of milk (ii) other conditions remaining the same, the higher the temperature of inlet air, the higher was the free fat contents of the powder (iii) between powder from cow's and buffalo's milk made under identical conditions, product from buffalo's milk always registered higher free fat compared to its counterpart from cow's milk (iv) the influence of higher preheating temperature on free fat content was similar with either types of milk and (v) influence of higher temperature of inlet air appeared to be more in the case of product from cow's milk than in the case of similarly made product from buffalo's milk.
ADSORPTION OF OXYGEN BY ROLLER DRIED WHOLE MILK POWDER DURING STORAGE.

1  •  80°C PRE-HEATING TEMP.  
2  •  95°C "  "  "  37°C
1  •  80°C "  "  "  30°C 
2  •  95°C "  "  "

FIG. 31 COW AIR PACK
FIG. 32 BUFFALO AIR PACK
FIG. 33 NITROGEN PACK
FIG. 34 NITROGEN PACK

OXYGEN %

STORAGE IN MONTHS
As observed earlier by Semkamer (1965) an increase in free fat reduces wettability and consequently results in poorer dispersibility of the powder.

**III - Effect of storage temperatures on the rate of oxygen absorption and peroxide development in whole milk powder under different conditions of packaging.**

The results relating to the rate of oxygen absorption in roller dried cow's milk preheated to different temperatures, packed with and without nitrogen gas and stored at temperatures of 30 and 37°C are presented in figure 31 and 33. The oxygen content of different packs have been calculated on the assumption that the average oxygen in head space of air packed samples was 21.0 per cent and in the head space gas of nitrogen packed tin was 3.2 per cent.

The results for the roller dried powder from cow's milk preheated to 30°C and stored at 30 and 37°C show that at the higher storage temperature the rate of oxygen absorption was more but at the end of twelve months, there was still some oxygen in the head space of all the samples packed in air. Between the two temperatures namely 30 and 95°C, rate of absorption was high in the samples corresponding to the low preheating temperature i.e. 30°C as compared to samples prepared from milk preheated to 90°C. This applied to samples stored at either temperature and packed in air or nitrogen.

The trend of results of buffalo whole milk powder (roller dried) was same for the samples of powder stored
ADSORPTION OF OXYGEN BY SPRAY DRIED WHOLE MILK POWDER DURING STORAGE

1  80°C PRE-HEATING TEMP.
2  95°C  "    "    "  37°C
3  115°C "    "    "    "    "    "  30°C

FIG. 35  COW AIR PACK

FIG. 36  BUFFALO AIR PACK

FIG. 37  NITROGEN PACK

FIG. 38  NITROGEN PACK

OXYGEN %

STORAGE IN MONTHS
at 30 and 37°C under the above specified conditions of packaging i.e. air and nitrogen (Refer Fig. 32 and 34).

Samples of spray dried powder from cow's milk preheated at 85°, 95° and 115°C and packed in air subsequently stored at 30°C showed complete absorption of oxygen content in course of five, eight and nine months respectively (Fig. 35 & 33). When the sample of powder prepared from similarly preheated milk packed in air and stored at 37°C, complete absorption of oxygen was recorded in two, five and six months of storage respectively.

Samples of powder from cow milk similarly treated packed with nitrogen and stored at 30°C showed that the samples preheated at 30°C had completely absorbed oxygen preheated to 95 and 115°C still had residual oxygen in the head space after the period of twelve months. When similar samples packed was stored at 37°C, the oxygen in head space of sample from milk preheated to 85°C disappeared completely in 10 months while the oxygen in packs corresponding to samples of milk preheated to 95 and 115°C did not show complete disappearance of oxygen at the end of twelve months storage period.

In case of samples of spray dried powder manufactured from buffalo's whole milk preheated at 80, 95 and 115°C packed in air and stored at 30°C showed a complete absorption of oxygen in four, six and eight months, whereas, in case of samples stored at 37°C, the complete absorption
of oxygen was in two, three and six months corresponding to their preheating temperatures of milk. Data are presented in Fig. 36 and 38.

The samples packed in nitrogen gas and stored at 30°C showed presence of oxygen in the head space in all the samples of whole milk powder manufactured from milk preheated at 90, 95 and 115°C. Whereas, in case of samples of milk preheated at 80°C and stored at 37°C, the absorption of oxygen was found complete at the end of eleven months. Samples of milk powder prepared from milk preheated at 95°C and 115°C did not show complete disappearance of oxygen from the head space even at the end of twelve months storage at 37°C.

Since amount of oxygen absorption is a fair index of rate of deterioration of whole milk powder, it may be concluded that preheating at higher temperatures prevents earlier spoilage of spray dried whole milk. Between spray dried and roller dried whole milk powders, roller dried powder appeared to have greater resistance towards the absorption of oxygen especially when air packed. Gas, packing, though not up to the standard in present studies, helped retard oxidation to a considerable period especially at higher storage temperature.

The above observation regarding the effect of preheating temperature, nature of pack, and storage temperature applied generally to the product of both cow's and buffalo's milk. However, the product from buffalo's milk
FIG. 39. DEVELOPMENT OF PEROXIDE VALUE DURING STORAGE OF ROLLER DRIED WHOLE MILK POWDER

- 80°C PRE-HEATING TEMP. 37°C
- 95°C
- 80°C
- 95°C

COW
AIR PACK

BUFFALO
AIR PACK

PEROXIDE VALUE MIG EQUIVALENT / KG.

STORAGE IN MONTHS

NITROGEN PACK
NITROGEN PACK
had relatively lesser resistance towards the oxidation as compared to its counterpart made from cow's milk under identical conditions of packing and storage. It might be due to less release of -SH group at preheating temperatures of 30 and 35°C.

**Peroxide value**

The results relating to the rate of peroxide development in roller and spray dried of cow's and buffalo's milk preheated to different temperatures, packed with and without nitrogen gas and stored at temperatures of 30 and 37°C are presented in Fig. 39 and 40.

The results show that rate of peroxide development was more in spray dried powder as compared to roller dried powder at both the storage temperature i.e. 30 and 37°C. In all the cases, an induction period was noticeable after which the increase in the peroxide value was rapid. In case of samples packed in air, all the samples showed a more rapid development of peroxides than those packed in nitrogen. It was also observed that powder prepared from milk preheated at 80°C gave rapid development of peroxide as compared to samples manufactured from milk preheated at 95 and 110°C.

The same trend of peroxides development was found in samples manufactured from buffalo's milk. It was observed that peroxide value of 2 corresponded to the stage of discernable oxidation in the product oxidised flavour.

From above results it is clearly seen that the powder prepared from the milk preheated at the higher temperature remained in good condition, on an average, for twice, as
FIG 40 DEVELOPMENT OF PEROXIDE VALUE DURING STORAGE OF SPRAY DRIED WHOLE MILK POWDER.

1. $80^\circ$C PRE HEATING TEMP.
2. $95^\circ$C
3. $115^\circ$C

\[
\begin{align*}
\text{Cow} & \quad \text{Buffalo} \\
\text{37$^\circ$C} & \quad \text{30$^\circ$C}
\end{align*}
\]

**AIR PACK**

**NITROGEN PACK**

Storage in months
long as the powder prepared from milk preheated at the low temperature, the effect of the higher pre-heating temperature being to extend the induction period before rapid deterioration began. This improvement is due to (a) more efficient destruction of oxidizing enzymes originally present or produced in the milk by the growth of micro-organisms (b) the production in milk of sulphhydryl compounds by the action of heat on the proteins, particularly the lactalbumin and the protein associated with the fat globules. Holm et al (1926) reported that high preheating temperatures helps to prolongs the keeping quality of whole milk powder.

The relation of storage temperature to keeping quality of whole milk powder packed in air studied by Dahle and Palmer (1924) showed that within a temperature of 4 to 20°C showed scarcely any deterioration after one year while powders air packed in identical containers but stored at 37°C had suffered pronounced deterioration after three months in storage. Lea, Moran and Smith (1943) found that the effect of gas-packing and storage on palatability of whole milk powders and concluded that powder containing up to 3 per cent oxygen in the free space gas of can could be kept in good condition for 2 years at 37°C and for several at 15°C.
IV. Effect of storage temperatures on physical characteristics of whole milk powder (roller and spray).

Milk powder in hermetically sealed packing is safe against reabsorption of moisture under favourable conditions and, therefore, the moisture content in powder did not find.

Colour:

There was no change in original colours of either type of powder at the storage temperatures of 30 and 37°C. The reason was that the moisture levels in powder were below 5 percent. Coulter et al. (1943) reported that milk dried to moisture levels below 5 percent showed essentially no change in colour even during a 2 year storage at 37°C.

Solubility index:

It was found that there was no appreciable change in the solubility index of either types of powder during storage.
Observations on casein micelle in skimmed/pre-concentrated/reconstituted milk under electron microscope (cow and buffalo)
SECTION V

OBSERVATIONS ON CASEIN MICELLE IN SKIM MILK
RECONSTITUTED FROM DILUTED MILK UNDER
ELECTRON MICROSCOPE

Materials

Solutions of skimmed, preconcentrated and reconstituted milk were prepared in Experimental Dairy, Dairy Technology Division.

Method

Following method suggested by Carroll et al. (1968) was adapted to observe casein micelle of milk under electron microscope.

1) Fix the skimmed, preconcentrated and reconstituted milk (preconcentrated milk - 45 per cent total solids) was diluted to the original composition of milk) in a 1.0 per cent glutaraldehyde solution for 15 minutes. The recommended volumes were 0.2 ml of skimmed milk to 2 ml of fixative.

2) Dilute the fixed skimmed milk with distilled water (recommended dilution is 1:50 to 1:100). In present study 1:50 dilution was found to be best.

3) Put a drop of diluted material on sterilized copper grids coated with formvar film and dry in air. Excess solution blot off with filter paper.

4) Shadow casted with gold-platinum alloy at an angle of about 30-25°.

5) It was examined under electron microscope (Model Philips 100) and photographed at 5000 magnification.
Plate I.
Electron microphotograph of Casein Micelle in cow's skimmed milk (x 5000)

Plate II.
Electron microphotograph of Casein Micelle in buffalo's skimmed milk (x 5000)

Plate III.
Electron microphotograph of Casein Micelle in cow's skimmed pre-concentrated milk (x 5000)

Plate IV.
Electron microphotograph of Casein Micelle in buffalo's skimmed pre-concentrated milk (x 5000)
Plate-V. Electron microphotograph of Casein Micelle in cow's skimmed reconstituted milk (x 5000)

Plate-VI. Electron microphotograph of Casein Micelle in buffalo's skimmed reconstituted milk (x 5000)
OBSERVATIONS ON CASEIN MICELLE OF SKINNED CONCENTRATED (SKINNED) AND RECONSTITUTED (SKINNED) OF COW'S AND BUFFALO'S MILK UNDER THE ELECTRON MICROSCOPE

It is seen from plates I and II that the size of casein particles in buffalo's milk are bigger than that of casein particles of cow's milk. It was difficult to measure size of casein particles, as the electro-micrographs were taken at 5000 magnification (recommended magnification is 20,000 to 30,000).

Concentration of same samples of milk to 45 per cent total solids in a vacuum pan under commercial practice did not appear to cause any change in the size of casein in the either types of milk (Refer plates II and IV).

When milk was prepared by reconstitution of skimmed milk powder from cow's and buffalo's milk, it is observed that samples reconstituted from buffalo's milk powder exhibited clustering of casein to a greater extent than samples reconstituted from cow's milk powder (Plates V and VI).

Instantised skim milk powder from buffalo's milk when reconstituted appeared to yield a product where there was evidence of casein clustering to greater extent than similar clustering in the counter part from normal (non-instantised) skimmed milk powder from buffalo's milk.

The above observations seems to confirm the commonly held views that the casein in buffalo's milk is different
late-VII. Electron microphotograph of Casein Micelle in buffalo's (instant) reconstituted milk (x 5000)
from casein in cow's milk and that heat damage on buffalo's milk casein (in process such as spray drying) is more intense as compared to similar effects on casein of cow's milk.

Galspaj (1962) investigated with electron microscope, the casein micelle in reconstituted cow milk from spray dried and roller dried powders. Casein from roller-dried milk had coalesced completely whereas casein from spray dried milk still appeared mainly as discrete, more or less spherical particles.
Summary and Conclusions
SUMMARY AND CONCLUSIONS

Studies on the physical characteristics of buffalo's and cow's roller, spray dried milk powder, have been carried out, separately on the products from skimmed and whole milk. Production of instant non fat milk powder has also been included.

The importance of the study undertaken and its industrial significance has been discussed.

Available scientific information on the topic from the literature has been reviewed in general and with particular reference to the physical characteristics of milk powder i.e. solubility, wettability and sinkability, bulk density and colour as influenced by the manufacturing processes and storage conditions. The size of casein particles in skimmed milk and effect of heat on casein particles in concentrated and dried milk have also been reviewed.

Scope and plan of work has been explained to include.

1) Manufacture of Roller dried skimmed and whole milk powder using varying levels of preheating temperature of milk and steam pressures in rollers.

2) Manufacture of spray dried skimmed and whole milk powder using varying levels of preheating temperature of milk and inlet air temperatures in the spray drying plant.

3) Test packed of product packed in poly-kraft and polythene bags to simulate whole sale and retail packaging conditions and storage at room temperature (10-35°C) have been indicated.
Whole milk powder was packed with and without nitrogen gas in 400 g. tins. Temperature of storage of skimmed milk powder varied from 10° to 35° C and relative-humidity was from 50 to 90 per cent during the period of study. Whole milk powder was stored at the temperatures of 30 and 37° C for 12 months.

4) Showing of the effect of solids in concentrated milk on the quality of normal spray dried skimmed milk powder.

5) Studies on the viscosity, size and number of lactose crystals in concentrates stored at different temperatures for different time intervals.

6) Production of instant non-fat milk powder from 50 per cent total solids in the concentrate at different atomizer speeds.

The results of the investigation on the above aspects are summarised below:

Roller dried skimmed milk

Preheating temperature had no effect on solubility, wettability and dispersibility of roller dried milk powder. However, the high pre-heating i.e. 95°C (temperature) was found to affect the colour of powder slightly.

Steam pressure of 80 p.s.i. had considerable affect on solubility, wettability and dispersibility as compared to 60 p.s.i.

Solubility of buffalo's milk powder was found to be less as compared to cow's milk powder. However, the solubility of buffalo's milk powder was well within the limits prescribed by
A.D.M.I. i.e. 15 ml. Bulk density of either type of milk powder was found to depend on the grinding and size of powder particles.

The above mentioned physical characteristics of roller dried milk powder, were not appreciably affected during storage at 10° - 35°C in poly kraft bags. The moisture content of all stored samples was below 4.0 per cent. The moisture content of milk powder and storage temperature are principal factors to affect the colour, pH and above mentioned physical characteristics.

Spray dried skimmed milk

It was found that as total solids increased in concentrate, the moisture content, solubility, wettability, sinkability, dispersibility, particle size and bulk density increased. The reason is that bigger drops at the time of atomizing are formed as the total solids in concentrate increased. This affects the air content which in turn increased the bulk density and subsequently physical characteristics. However, the values of physical characteristics obtained in present studies were found to be low because of high atomizing speed i.e. 25,000 r.p.m. which was used because of necessity to run the plant to its full capacity. Moreover, the centrifugal atomizer gives trifle less values because of more air content in powder particles.

Preheating had no effect on physical characteristics of spray dried milk powder. However, it was found to affect
the colour of either type of milk powder. The colour of
buffalo's milk powder was found to be almost white and that
of cow's milk powder was yellowish.

The higher air inlet temperature i.e. 200°C affected the
moisture per cent of powder and solubility, wettability,
sinkability and dispersibility. There was no change in
colour. It was found that the size of powder particle was
less as compared to powder dried at 180°C inlet temperature.
The reason was that the feeding rate had to be increased to
maintain the outlet temperature at 95°C which was kept constant
with both the inlet air temperatures i.e. 180°C and 200°C. The
results regarding the bulk density and other physical character-
istics reported by earlier worker on cow's milk were quite
high. In the present study, the values were found to be low
because of high r.p.m. i.e. 25,000 and the centrifugal type of
the atomizer.

During storage for 12 months there was slight change in
moisture, solubility, wettability and dispersibility. Though
the moisture content was below 3.0 per cent in all samples,
the moisture content increased because of absorption of
moisture from atmosphere as the powder was packed in poly kraft
bags and stored at room temperatures i.e. 10°C - 35°C.

Solubility index was slightly more, wettability, sinka-
bility and dispersibility low of buffalo's milk powder as
compared to cow's milk powder. It may be due to the lesser
heat stability of buffalo's milk.
Studies on viscosity, number and size of lactose crystals in pre-concentrated milk for instantization

Prior to manufacture of non-fat buffalo's instant milk powder, laboratory trials were conducted to find the most suitable total solids percentage in concentrate, temperature of holding and time to crystallize lactose. Crystallization helps in increasing the viscosity which in turn helps in manufacturing the good quality of instant milk powder in single pass method. In laboratory trials, it was found that the 50 per cent concentrate, 5°C temperature of holding for 12-14 hours was the best to manufacture the instant milk powder, since the 50 per cent concentrate had initial viscosity as compared to 40 per cent concentrate. During storage at 5°C for 12-14 hours, the viscosity was doubled. The number of crystals and size of crystals were found to be considerably high as compared to 40 per cent concentrate. Hence the 50 per cent concentrate, 5°C holding temperature and 12-14 hours holding period was selected to manufacture the instant milk powder from buffalo's milk.

Production of near instant skimmed buffalo's milk powder.

While manufacturing the instant, the low r.p.m. i.e. 7,000, 10,000 and 15,000 were used. It is established fact that low r.p.m. of atomizer gives the bigger powder particles with more enclosed air which in turn gives more bulk density, wettability, sinkability and dispersibility.

It was found that the moisture, particle size, particle density, wettability, sinkability and dispersibility increased
as the r.p.m. of atomizer decreased, whereas the solubility index was found to decrease since the residence time in drying chamber was same and the quantum of heat received by all powder particles corresponding to each atomizer speed was same. At the lower speed, the droplet size is always bigger, so the moisture content, particle size and particle density is more which in turn gives more bulk density, wettability, sinkability and dispersibility. Among the various speeds of atomizer used, 7,000 r.p.m. was found to be best to manufacture good quality instant powder.

Change in physical characteristics of instant milk powder during 24 weeks storage period at 28-30°C was found very negligible. It was also found that the moisture content was increased but it was well within the standard limit i.e. 5.0 per cent at the end of storage period.

**Roller dried whole milk**

Preheating temperature had little effect on solubility. It has been reported by number of workers that high pre-heating improves considerably the keeping quality. High preheating temperature gave cooked flavour particularly at 95 and 115°C.

High preheating temperatures increased colour intensity in either type of powder. At pre-heating temperature of 80°C, the colour was found to be almost white in buffalo's milk and that of cow's was yellow because of carotene content. Higher preheating temperatures gave brownish tinge to powder.
At high steam pressure i.e. 80 p.s.i. the solubility index was found more in either type of powder. Solubility index of buffalo's milk powder was more as compared to cow's milk powder. It was also found that the free fat in powder was found more when prepared with high steam pressure in the rollers.

Since, the roller dried whole milk powder had less solubility and about 80 to 90 per cent free fat, the wettability and dispersibility could not be determined. Bulk density of either type of powder overlapped but the values were less as compared to corresponding skim milk powder because of their fat content.

High preheating temperature prolonged the keeping quality of either type of powder. It may be due to the production of sulphhydryl groups which act as antioxidants.

Gas packed samples showed good keeping quality over air packed. Upto one year, no appreciable changes were found in gas packed samples. Oxygen content, flavour and peroxide value were found to be correlated as oxygen content decreases in head space during storage, the flavour and peroxide value increased.

High preheating temperatures imparted perceptible brownish colour and cooked flavour to either type of powder. However, these defects were more than off set by the increase in keeping quality resulting from such treatment.
Spray dried whole milk

High preheating showed little change in solubility. High inlet temperature was found to increase solubility index and free fat of powder.

Free fat was found to be major factor affecting the physical characteristics of powder. Because of free fat the wettability was found to be low, as fat repelled the water at the time of contact. The wettability, dispersibility and bulk density of either type of powder were found to overlap. Due to free fat, it was very difficult to find out correct values for dispersibility. However, high solubility index was found in buffalo's milk powder but it was well within the limit prescribed by I.S.I.

Bulk density was found to be low in either type of powder as compared to skim milk powder because of their fat content.

Preheating at 95 and 115°C showed prolonged keeping quality as compared to preheating at 80°C. High preheating doubled the keeping quality of air packed samples stored at 30°C.

All gas packed samples from milk, preheated to 80°C showed 12 months shelf life at 30°C and 10 months at 37°C. Higher preheating temperature showed more than 12 months shelf life of powder. During storage period, the oxygen content in head space was found to decrease.

Solubility was found to be very little affected.
Structure of casein micelle under electron microscope

Roller, Spray and Instant dried skimmed/whole buffalo's milk have been examined under microscope. In general the structure of powder from buffalo's milk appear to be similar to the structure of powder from cow's milk. The size of casein particles of buffalo's milk are bigger than that of casein particles of cow's milk. Heat damage of buffalo's milk casein (in process of spray drying and instant milk powder) was more intense as compared to similar effects in casein of cow's milk.

CONCLUSIONS

1) In general the physical characteristics of skimmed/whole buffalo's milk powder made by different processes corresponded to the similar quality of products from cow's milk. However, solubility index of buffalo's milk powder was slightly higher than that of cow's milk powder. Data on solubility, dispersibility, wettability and sinkability showed that the value for the two types of powder overlap. There was slight difference in the free fat of two types of whole milk powder.

2) Keeping quality of skimmed buffalo's milk powder corresponded to the keeping quality of skimmed cow's milk powder under identical condition of packaging and storage.

3) In the case of whole milk powder high preheating temperature of milk and packaging of powder in nitrogen gas had similar beneficial influence to prolong the keeping quality of buffalo's milk powder as in case of cow's milk powder.
4) Manufacturing sections such as preheating temperatures and steam pressure in rollers in the case of roller drier, inlet and outlet temperature of air in the case of spray drier, all influenced the quality of buffalo's milk powder in the same way as for powder from cow's milk.

5) It is feasible to produce near instant skimmed milk powder using buffalo's milk by the single pass process prescribed for the anhyd dairy equipment. The dispersibility of product was recorded to be about 88 gr. i.e. well within the specifications of A.D.M.I. The optimum condition for production of near instant powder appeared to be:
   a) 50 per cent total solids in concentrate
   b) 50°C holding temperature for 12-14 hours for crystallization of lactose.
   c) 7,000 r.p.m. of homogenizer speed
   d) 200°C inlet air temperature and 95°C outlet air temperature.

6) Size of casein micelle of buffalo's milk are bigger as compared to casein micelles of cow's milk. Effect of heat on casein micelle is indicated to be greater in buffalo's milk powder and instant powder as compared to cow's milk powder. There is more clustering of casein micelle in buffalo's milk powder.
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