

**STUDIES ON THE MANUFACTURE OF
INSTANT CHOCOLATE MILK POWDER
FROM BUFFALO MILK**

By
SHASHIDHARA T.S.

**DIVISION OF DAIRY TECHNOLOGY
NATIONAL DAIRY RESEARCH INSTITUTE
(I. C. A. R.)
KARNAL (Haryana) INDIA**

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STUDIES ON THE MANUFACTURING OF INSTANT CHOCOLATE
MILK POWDER FROM BUFFALO MILK

A DISSERTATION

SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS
FOR THE DEGREE OF
MASTER OF SCIENCE

IN
DAIRYING
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By
SHASHIDHARA, T.S.
DAIRY TECHNOLOGY DIVISION

NATIONAL DAIRY RESEARCH INSTITUTE
(I.C.A.R.)
KARNAL (Haryana)

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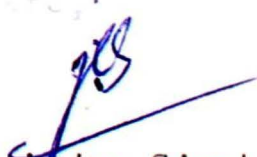
Dr. Joginder Singh,
B.Sc.(Hons), M.Sc.(Tech.), Ph.D.
Scientist (S-2)
Division of Dairy Technology,

DAIRY SCIENCE COLLEGE
NATIONAL DAIRY RESEARCH INSTITUTE
(I.C.A.R.)
KARNAL (Haryana)

Dated 27th May, 1982

This is to certify that Shri Shashidhara, T.S. of the National Dairy Research Institute, Karnal, participated in the planning of this study, carried out the experimental work involved, analyzed the data and prepared this report on "STUDIES ON THE MANUFACTURING OF INSTANT CHOCOLATE MILK POWDER FROM BUFFALO MILK".

He did these in partial fulfilment of the requirements for the degree of MASTER OF SCIENCE In DAIRY TECHNOLOGY of the Kurukshetra University, Kurukshetra, under my supervision. Help and assistance given by individuals as well as Institutions in the prosecution of the work has been suitably acknowledged.


(Joginder Singh)

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Shashidhara T.S

(Shashidhara, T.S.)

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INTRODUCTION

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CHAPTER - I

The first part of the report deals with the general aspects of the subject. It covers the history of the product, its composition, and its uses. The second part of the report deals with the specific aspects of the subject. It covers the production process, the quality control, and the packaging. The third part of the report deals with the marketing aspects of the subject. It covers the distribution channels, the pricing, and the promotion. The fourth part of the report deals with the future prospects of the subject. It covers the new products, the new markets, and the new technologies.

are used in the production of

INTRODUCTION

of chocolate flavoured milk. The product is a combination of chocolate and milk. It is a popular product among children and adults. The product is available in various forms such as liquid, powder, and bars. The product is used as a snack, a dessert, and a beverage. The product is also used in various recipes such as cakes, cookies, and ice cream. The product is a good source of energy and nutrients. It is also a good source of calcium and iron. The product is a healthy and delicious product.

1.0 INTRODUCTION

Modern dairy industry in our country is dependent upon buffalo milk constituting about 60% of the total milk production which could be partly utilized during flush season for the manufacture of milk powders including chocolate milk powder. Whereas skim milk and toned milk could be diverted for milk powder making, the surplus fat resulting from the separation of buffalo milk could be profitably utilized for the manufacture of high fat products like butter and ghee.

In advanced dairying countries, a considerable quantity of market milk is presently used for the manufacture of flavoured milks. It readily finds preference among those consumers who do not have taste or palatability for market milk. Further, the flavoured milk provides nutrients in adequate quantities for the growing children and adults. Among the different flavours that are used in the preparation of flavoured milks, cocoa or chocolate flavour finds preference among children and upto some extent in adults as well. The liquid chocolate flavoured milk has certain drawback such as lower keeping quality, a tendency to form sediment and cream plug on storage. Also there are problems which

are inherent with the marketing and distribution of chocolate milk because of the breakage of bottles, spillage from single service containers and disposal of returned chocolate milk.

To overcome the above drawbacks some commercial milk factories in the Western countries have started manufacturing chocolate milk in powder form, from cow milk, which on reconstitution gives satisfactory acceptability. At the same time, the dried product can be stored and transported conveniently with satisfactory keeping quality.

In India, especially in some of its big cities, the sale of flavoured milk is catching up very fast. Although the use of chocolate is no doubt expensive, yet due to the superiority of its flavour to other synthetic and natural flavours, its potential isⁿ boosting the sale of chocolate milk powder merits further investigation. Further, since refrigeration facilities are not available in most of our Indian houses, the longer keeping quality of chocolate milk powder at atmospheric temperature will substantially add to its popularity.

Regular chocolate milk powder is not well accepted by the housewife for beverage purposes due to the difficulty with which it reconstitutes in cold water.

Further, it has got a poor pouring characteristic and a tendency to cake and dust. These drawbacks of regular powder led to the advent of 'Instant' chocolate milk powder. Scientific information on the manufacture of instant chocolate milk powder is very scanty and is mainly confined to that from cow milk. Buffalo milk behaves in a different way as compared to cow milk owing to its compositional differences and differences in its physico-chemical properties. The present study is, therefore, planned to standardise the method of manufacture of Instant chocolate milk powder from buffalo milk. The various physico-chemical attributes of the product will also be investigated. The information gathered, it is hoped would provide the necessary impetus for dairy factories to diversify their activities and products which in turn would bring profit to the industry.

2.0 REVIEW OF LITERATURE

Sufficient technological information on chocolate milk powder by spray dried method and also instant chocolate powder by single-pass instantization is not available in literature. The information collected on chocolate milk and dried milks has been reviewed under the following headings:

- 2.1 Different instantization methods for milk powder.
- 2.2 Physico-chemical characteristics of dried milk.
- 2.3 Chocolate milk and powder.

2.1 Different instantization methods for milk powder:

There are four broad categories under which most of instantization method fall. They are:

- 2.1.1 Rewetting method.
- 2.1.2 Single-pass method.
- 2.1.3 Surface active agents cum rewetting method.
- 2.1.4 Twin spray drying method.

2.1.1 Rewetting method:

David Peebles (1956) was the first to invent instant milk powder. The process (called rewet method)

consisted of controlled moistening, agglomeration and re-drying of regular spray dried milk powder referred to as the base powder. One of the earliest workers to develop commercial methods of instantization of milk powder was Corlson et al. (1956). The base powder was agglomerated in a horizontal chamber with moist air raising the moisture content to 6-10 percent. The wetted particles pass to a cyclone collector from which they fall into a moving stream of air heated to 132-149°C.

Lauder and Hodson (1958) got a patent for the manufacture of instant milk powder, which was also re-wetting by steam. The base powder was wetted with steam to a moisture content not exceeding 9.0% and preferably 5.5%. The resulting agglomerated moist powder immediately contacted a stream of hot air which quickly dried the agglomerated moist powder and carried it horizontally from the chamber. The instantized product contained relatively high β -lactose and low α -lactose fractions. This was thought to improve the dispersibility of powder. Bissel (1958) also got a patent on steam instantizing process. The powder was fed on to a moving belt from which it fell as a curtain into the path of steam jets. The steam wetted the powder and simultaneously projected it into a chamber through which second belt passes. It was claimed that a substantial portion of lactose in milk is agglomerated of particles into instantized powder.

powder present in B-anhydrous form was changed to the α -hydrate form in the instantized product.

In the system used by Griffin (1959) the instantizer was cone shaped and very compact. The base powder was made to fall as a curtain between two steam jets. The powder was instantly wetted approximately to 7% moisture and fell into the top of the instantizer where it encountered heated turbulent air. The turbulent air caused the powder particles to agglomerate into clusters and also removed the moisture previously picked up from the steam jets. The instantized product fell on to a moving belt which carried it away for cooling and packing. There was an arrangement for the return of unagglomerated powder.

Scott (1959) obtained a patent for a method where base powder was wetted in a hydration chamber with a fine spray of water or milk instead of steam to a moisture content of 10-14%. This was a droplet agglomeration instead of steam condensation. It then conveyed to a hammer mill during which times (15 to 20 sec.) the wetted protein swelled and the milk sugar hydrated and absorbed its water of crystallization. The purpose of hammer mill was to break up the large wetted chunks of powder which may form in the conveyor coming from the hydration vessel. Also, the blades in the mill caused the agglomeration of particles into clusters or granules

of the desired size. The powder was then re-dried on porous belts which travel through drying ovens at temperatures between 71 and 116°C. Shields (1962) also got a patent where a spray dried skim milk powder was moistened excessively with water to a moisture content of about 15%, held for a sufficient period to convert a substantial portion of the lactose present into the α -hydrate form, dispersed in a stream of air and mixed with normal skim milk powder particles in such proportions that the added powder provides about 25% of the total solids in final product. The resulting clumps were dried with hot air at a temperature rising from 82 to 127°C. The dried clumps had a moisture content of about 3.5% and were claimed to be highly soluble in cold water.

Boer (1972) obtained patent for a process in which freely flowing milk powder particles fall into a horizontal jet of moistening gas which forcibly deposits the moistened particles on to a vertical belt moving in an upward direction. The particles pass through a drying zone and are then scrapped from the belt. The resulting powder was claimed to be more soluble. Kleeman and Rothmayr (1974) got a patent for instantizing powdered product of tea, coffee, drinking chocolate and other beverages and foods by rewet method. Stream of powdered material was contacted with a saturated steam at 100 to 140°C and a stream of inert gas (at 10 to 40°C) such as

air, nitrogen or carbonic anhydride.

Jensen (1975) developed the rewet method agglomerates and prevent problems of dusting in edible dye-stuff, by the use of granulating disc. Granulating disc unit has short cylindrical side and adjustable scrappers to prevent product from accumulating on it. The powder is cascaded on to the pan and moistened with water from spray nozzle. The pan is inclined between 25 and 45 degrees to the vertical and rotated at 10 to 25 rpm. These parameters along with feed rates of powder and water determine particle size and ^{glu}stability. Panchal (1981) used the disc agglomerator to produce milk powder agglomerates in a laboratory scale experimental investigation.

Lerst (1976) set up an Experimental Plant to instantize 100 kg of SMP per hour. The process involved wetting of SMP by steam to 8-10% moisture, pre-drying and final drying to 4% in a fluidized bed type. The author observed that the quality of instantized product depended upon the quality of SMP and its manufacturing process. The steam required at 102 to 104°C was 17 kg/100 kg of product and the bulk density of final product was 0.15 to 0.25 g/ml.

Anhydro (1976) developed a fluidized bed instantizer containing a distribution plate for the passage of fluidization gas and three other sections for carrying out moistening, agglomeration, drying and cooling. Dornyei et al. (1978) established the advantages of fluidized bed over the pneumatic conveying. They studied the optimization for drying of skim milk agglomerated by rewetting. The suggested optimal parameters were: temperature of drying air 110-130°C, air flow 0.65 to 0.66 metre/sec., relative vapour content of air before heating 0.7 to 0.8%, screen loading 23 to 24 kg/sq.m, amplitude of vibration 2.4 to 4 mm, frequency of vibration 23.3 to 23.4/sec., initial moisture content 10 to 10.5%. Formulae for calculating the total drying time and also the average time spent by agglomerated in the fluidized bed were arrived at. In another patented method (Wahli, 1979) of agglomeration by rewet system, stream of dried skim milk particles and a moistening agent such as steam was projected on to a fixed obstacle, usually in a conical shape, and discharging the agglomerated product into a fluidized bed drier for final drying.

Nakazto et al (1980) developed an apparatus, suitable for producing readily soluble milk granules, consisting of vertical outer and inner concentric tubes of which had heat input at the bottom.

different length and diameter, partly overlapping each other with a circumferential gap between them. The powdered material released from a distributor travels down through the gap under the action of a whirling stream of compressed air.

2.1.2 Single-pass method:

Coulter and Townley (1959) had one of the earlier patents in this kind of driers. They could obtain large particle instant powder by varying conditions of drying. Agglomeration in single-pass was also brought about by bringing into contact partially dried particles with wet particles. Ammdson (1960) produced single-stage agglomerates with his Experimental tower drier by using five nozzles in certain arrangement. Four of the nozzles were arranged at a certain level radiating out 30.48 cm from a common centre and 90 degrees apart. The fifth nozzle was placed 0.91 m. below the centre of the other nozzle. This resulted in contact of mist of relatively wet particles from the lower nozzle to a mist of relatively dry particles from four upper nozzles. Arrangement of this type resulted in a powder having 6 to 7% moisture and thus further drying was necessary.

Sanna (1960) developed a unique tower drier which had heat input at two or more pre-determined

levels during drying. The falling milk particles pass through at least two heating zones, the first being heated by electro-magnetic wave energy and the second by radiant energy supplied by a series of infra-red lamps.

Alfa Laval (1961) has patented the equipment used in this method. In their system positioned centrally above the powder exit of the drying chamber is a suction element which evacuates the fines. These fines are then passed through a cyclone where they are separated and re-diverted into atomizer. The fines are dispersed through the tangential openings situated above the normal liquid discharge suction of the atomizer. The plant had a provision for the introduction of vapour or mist into the conduit through which the fines are returned into the drying chamber.

Of various methods in which fines can be re-introduced into drying chamber, the one where the fines are blown up under the atomizer wheel is the best (Masters, 1979). This gives a uniform contact between the wet droplets and the fines and also prevents fines from coming into contact with hot air. Fines do not have good re-constitution properties, but once they are in the presence of relatively wet particles, form agglomerates and show better re-constitution properties.

Niro Atomizer (1961) patented an agglomerating unit which can be coupled directly to the outlet of spray drier. The powder containing at least 6% moisture is agglomerated in the first section of 3-section tunnel, by moist air. The vibration prevents the fusion and also crystallization of some constituents, causes the particles to lose the tendency to stick together. Drying is completed in the third section. Fines are passed back into drying plant. The powder produced had approximately 0.55 g/ml density and plant had a capacity of 300-2000 kg/hr.

Troesch and Wilk (1964) described a vertical cylindrical spray drier having central drying air entry and a peripheral spent air outlet. The product to be dried was sprayed through a low pressure nozzle and the finished product leaves the chamber at the pointed bottom preferably falling out freely. An arrangement for separation and return of fines was there. The equipment was claimed to produce powder particles of size 60-2000 μm in diameter and a bulk density of 0.7 g/ml.

Sanna (1964) patented a method of single-stage process where concentrated skim milk having 50-75% moisture is dried by air at 121-232°C and exposing the dried particles to partial cooling to cause the re-transfer of some of the moisture from the humid atmosphere.

The moisture of the particles was raised to 2.5-3.5% which promote formation of agglomerates. The partial cooling was affected by introducing un-heated air at atmospheric conditions into the spray-dried mixture by passing the mixture through a heat exchange zone.

The re-wetting method described earlier under 2.1.1 involved base powder to be re-processed. This re-processing of powder increases the cost of final product and in some instances may have a detrimental effect on flavour and solubility.

Sorensen (1973) reported that in the single-pass method the particles in the initial drying itself are obtained in larger particles by adjusting the spray drying conditions. These conditions were viscosity of feed, nozzle size, feed pressure and drying temperature etc. To obtain larger particles, the viscosity of feed to the atomizer should be increased either by increasing total solids or by lowering the feed temperature. Atomizer speed in the case of centrifugal atomizer or pressures in the case of nozzles have to be low. As the particle sizes were large and the rate of evaporation slower, the residence time of the particles had to be increased resulting in a design of tall drying chambers called 'Tower dryers'.

Jensen (1975) reported that the returned fines in a single-pass system may be introduced around the rotating atomizer or through a central pipe from below the atomizer, throwing the fines upwards against the atomized particles. The latter arrangement was considered to be better as it prevented excessive exposure of fines to hot air.

In the single-pass methods mentioned above, the powder particles have got a high moisture so that there could be agglomeration of the particles. But one disadvantage was that the moisture was in the inner spaces, while surface was dry, which was just the opposite of what is needed for agglomeration. This was one reason why single-pass method could not cause as good agglomeration as the re-wet method.

2.1.3. Surface active agents cum re-wetting method:

Peables (1959) patented a method in which skim milk was first spray dried and then instantized by re-wet method, and the warm powder was allowed to fall through a chamber in which melted milk fat was finely atomized. It resulted in absorption of fat into the porous structure of the agglomerated skim milk powder.

Sjollema (1960) in his method has mixed warm powder with the liquid surface active agent such as monoglycerol, and later dried in a spray drier. The final moisture content of 1.5 to 3.0%.

oleic acid or soyalecithin in an amount of 2% by weight for about 5 min. and then cooled. The product was claimed to show good instant properties in cold water even after one year of storage provided the lactose is not allowed to crystallize and thereby de-emulsify the milk fat. In a similar patented method McIntire and Loo (1964) opted for incorporation of lecithin in the range of 0.2 to 1.5% of the final product. The excess moisture from the agglomerates was removed outside the treating zone. The equipment could be used for manufacture of instantized and chocolate flavoured malted milk products.

Spilman and Nava (1964) in a method to manufacture instantized powder had fed concentrated milk, liquid lecithin and lactose seed crystals through centrifugal atomizer into a drying chamber. The particles were exposed to drying gas in such a way that they adhere to each other while still in moist condition. The lecithin in the final product ranged from 0.17 to 0.6%. Shields and Kempf (1965) patented a process where whole milk concentrate was seeded with lactose crystals and was then spray dried to a moisture content of 7 to 12%. The product was then held on a scroll conveyor, simultaneously intermixing with lecithin dispersed in a suitable liquid medium, and later dried in a shaker type drier to a final moisture content of 1.5 to 3.0%. The final product was claimed to have instantized properties.

In a process developed by Mourey (1965) the importance of milk fat fractions was brought into light and wettability of lower melting fraction of milk fat was shown to be better than that of high melting fraction. In their method, fractionation of fat was carried out before drying of the milk concentrate and the high melting fraction was re-cycled to the milk concentrate before it was spray dried; the lowmelting fraction being applied as a coating on the dried powder.

Williams and Busch (1966) obtained instant whole milk powder by homogenizing lecithinated milk fat with skim milk concentrate and then spray drying. The dissolving of lecithin into milk fat was at the rate of 1.1% and at a temperature at which milk fat is in a liquid state. The mixing of milk fat and concentrated skim milk was such that the fat was 6 to 48% in the final mixture and lecithin about 0.05 to 4.0%. The drying of moisture was either by conventional tower drier or by horizontal spray drier.

Baldwin and Sanderson (1972) described a method in which 0.6% lecithin (w/w) was applied in a straight through system. They also found that along with the quantity of lecithin added, the size of particles in the final product was also critically important. They proposed that particles less than 90 μm in size should

be less than 15% of the total product so as to have better instantization properties. Pisecky and Westergaard (1972) also developed a similar system which was used for commercial production.

Baldwin and Sanderson (1973) studied various types of surfactants like glycerol monostearate 1:1 ratio of A₊omos 300 and Tween 80, Span 80, control IP, Centrolene S. and Centrolene H. The methods of co-drying skim milk and cream mixed with surfactant, and mixing of dried agglomerates with surfactant dissolved in a solvent, were used for studying the above mentioned surfactants. It was observed that use of water as carrier for surfactant involved a drying stage to remove the moisture, in addition to requiring more concentration of surfactant.

Piscky and Westergaard (1973) observed that for best results lecithin should be added @ 1-3% of final dried product and of 15-25% of free fat in liquid form at ambient temperature. The minimum thickness of coating should be 0.1 μ m.

Jensen (1975) described a system using two vibro-fluidizers along with the process of lecithin coating. The powder was fed to the first vibro-fluidizer where hot air is blown through so as to make

or sprayed simultaneously in multi-nozzle

instant systems. Though single-

the powder warm and to remove fines that are formed by mechanical agitation during transport. Before the powder is fed to the second vibro-fluidizer the agglomerates are treated with lecithin delivered through a two fluid nozzle. The second fluidizer equalises the lecithin coating of all agglomerates. Sanderson (1978) was of the opinion that amongst the various methods used for manufacture of instantized whole milk powder, re-wet method will not be suitable since it gives rise to higher level of free fat and makes the powder more susceptible to **oxidation** due to second drying.

Rewet and single-stage methods were successful in agglomerating skim milk powder, but failed to get reasonable amount of agglomeration with whole milk powder. This was mainly because of hydrophobic nature of milk fat and its distribution over the surface of the particles.

The difficulties in instantizing of fat-rich products was solved, among other methods by the powder particles with surface active agents, whose role is to reduce the surface tension and permit the entry of water more easily, and to spread on the surface of particle to reduce the hydrophobic nature of the surface fat. There are numerous variations in applying surface active agents. It is applied along with wetting agent in rewet method, or mixed with skim milk concentrate before drying or sprayed simultaneously in multi-nozzle single-stage instant systems. Though single-pass method is suitable,

a second stage drying should be involved instead of drying large particles in the first drying chamber itself. This not only permits a lower outlet air temperature in main drying chamber but also reduces damage to powder caused by case hardening and heat denaturation. Also fluidised bed driers are most suitable than pneumatic secondary driers. Hensen et al. (1979) produced a process using Niro-Atomizer Spray drier and Anhydro fluid-bed equipment divided into three sections. The first section dried the powder to 2.5% moisture with air at 80°C and in second section 0.4% lecithin was added in the form of 45:55 mixture with butter oil, at 45°C. The third section had a stream of air at 45°C to distribute lecithin over the surface of the particles.

Fitzpatrick and Lweis (1979) got a patent for the production of instantized dried milk particularly milk with greater than 10% fat. The process comprised of wetting the dried milk to form a coating of ~~0.1~~ 0.5% lecithin (in relation to the wt. of agglomerates) by spraying with aqueous solution and subsequent drying in a fluidized bed drier.

2.1.4 Twin spray drying method:

Bauer and Marks (1969) and Lipatov and Kheitonov (1972) have described the twin spray drying method.

In this method, two fluids of different properties are spray dried simultaneously so as to get ~~S~~ertain desired properties. To get improved agglomeration properties, lactose crystals are seeded into the concentrates. But the technique of twin spray drying is being utilized where lactose solution is spray dried simultaneously with whole milk concentrate. In another combination skim milk concentrate is simultaneously spray dried with cream or butter oil. With both cream and butter oil, surfactants were used.

In one of the other non-conventional systems, agglomeration of powder was achieved by deposition on the drier wall and the powder then being removed by air jets (Peebles et al, 1964).

2.2 Physico-chemical characteristics of dried milk:

Physical characteristics of dried milk viz. density, solubility, dispersibility, wettability, sinkability and the chemical characteristics including moisture, fat, free fat and HMF are reviewed below:

2.2.1 Physical characteristics of dried milks:

2.2.1.1. Density:

Webb and Hufanagel (1943) reported that with the increasing degree of concentration of milk, the bulk

density of milk powder increases. These findings were confirmed by Manus and Ashworth (1948).

Syollema (1963) distinguished between minimum and maximum bulk density, corresponding respectively to the lowest and densest packing of the powder. Lea et al (1945), Lea and Game (1946) and Pallansch (1963) reported that particle density of milk powder is influenced principally by the amount of entrapped air. Free density referred to the air free solids. The values for true density of non-fat dry milk were found to be 1.44-1.48 g/ml. While for whole dry milk these were 1.26-1.32 g/ml.

Harper et al (1963) reported that maximum ease of dispersion for whole milk powder was achieved at a bulk density of about 0.36 g/ml and dispersibility decreased slowly as this value increased to 0.58 g/ml and rapidly beyond this value. Hall and Hedrick (1971) reported that the density of dry milk had three distinguished groups - bulk (apparent) density, particle density and the density of dry solids. Bulk density referred to the weight of dry solids, per unit volumes of the powder expressed in grams per ml. It is further divided into (i) packed and (ii) loose density. Packed density is determined after the sample has been tapped or vibrated

until the volume is relatively constant. Loose density is determined prior to vibrating or tapping.

2.2.1.2 Solubility:

First stage in the solubility of milk powder after its initial contact with water, is the wetting of particle surface (Mohr, 1961; King, 1971). This is followed by water absorption and swelling of proteins and soluble constituents (Mures and House, 1962). But, the smooth progress of reconstitution process was mainly dependent upon manufacturing and storage conditions which affected the nature of powder particles.

Abbot and Waite (1962) suggested that the solubility test method should take into account all the three characteristics of wettability, dispersibility and solubility. However, a wettability test complied with a solubility measurement for day-to-day factory control, both giving results quickly would provide useful information (Sat Prakash, 1969).

Mol and Jansen (1974) reported that the solubility of dried whole milk depends on particle size, the state of fat in dried milk and the drying temperature. For improved solubility the authors suggested the stage drying using fluidized bed in second stage.

Jensen and Hensen (1974) found that in the case of skim milk powder the solubility was influenced by the degree of concentration. An increase in the total solids of concentrate from 32.1 to 47.6% resulted in a deterioration of the solubility index from 0.02 to 0.32. No similar observation was found in the case of whole milk powder as measured immediately after production.

Baldwin and Woodham (1978) stated that the cause of poor solubility of skim milk powder might be due to increased protein content of dried skim milk, indirect heating, lower whey protein nitrogen index, high - temperature holding of the concentrate prior to drying, decreased particle size, packing at more than 76°C or combination of any of these factors.

2.2.1.3 Dispersibility:

Although, larger particles of dry milk are generally recognised as necessary for good dispersibility, exceptions have been observed by Konston et al (1965) . The very large particles of foam spray dried whole milk have less dispersibility than the small ones.

Danish et al (1966) reported that dried skim milk with improved dispersibility, bulk density and flavour properties could be obtained by coating the warm (100-140°F) dried milk particles with 0.033-0.66 percent active

lecithin based on the weight of total solids preferably in a vegetable oil solution at 110-180^oF. The product was then agglomerated by moistening and drying in an instantizer.

Tamsma et al (1967) described that high bulk density powder (non-fat dried milk) with good dispersibility could be obtained by adding surfactants to the concentrate and spray drying under conditions producing particle aggregation.

Hall and Hedrick (1971) pointed out that for good dispersibility an important consideration is the total heat treatment of the casein during processing. As the degree of heat application increases with perspective increase in ~~the~~ total solids, it brings about larger amounts of irreversible denaturation. Under the prevailing normal conditions of reconstitution procedures, this denatured casein does not form a stable dispersion.

Baldwin et al (1978) described that the dispersibility was adversely affected by the increased protein content of dried skim milk, indirect heating, lower whey protein nitrogen index, holding concentrates at high temperature before drying, decreased particle size, and packaging at more than 76^oC. Combination of any of these factors are likely to produce powders with poor solubility.

Stone et al (1954^a) described a mechanical low energy method for determining ease of dispersion of milk powder which utilizes low energy mechanical stirring and permits measurement of ease of dispersion of both non-fat and whole milk powders.

The ease of reconstitution on low energy mechanical stirring was measured by Stone et al (1954^b) by treating 52 g. of dried milk with 400 ml water in a mixer at 54 rpm for 10, 20, 30 and 40 seconds, draining off the milk through a 210 micron screen and determining the total solids in aliquot. The total solids dispersed in grams were taken as dispersion value.

Berg^{so}re and Buck Anderson (1956) determined the instancy of dried skim milk by stirring 5 g powder with 50 ml water for 5 seconds using a stirrer at 370 rpm. The solution was filtered through a weighed filter in a Buckner funnel and the solids left undissolved were determined by weighing the filter paper after drying.

2.2.1.4 Wettability:

Hall and Hedrick (1971) stated that wettability is the rate at which a mass of milk powder is penetrated by water. The tendency of dry milk to form lumps upon addition to water indicates lack of wettability. In general, large particles more irregular in shape provide

more space in the interstices for wetting rather than the small particles of symmetrical shape which enhance close packing of particles and thus inhibit penetration of water.

Coulter et al (1951) stated that the amount and dispersion of fat affected wettability. The authors reported no differences in wettability of dry milk with fat between 18-32%. Hibbs and Ashworth (1951) added 0.05% emulsifiers (Sorbitan monostearate, polyoxyethylene sorbitan monostearate and glycerol monostearate) to the milk before drying for achieving improved wettability of whole milk powder and to prevent churning of fat during re-combination but only a little had been achieved in controlling of the churning with a combination of 0.05% sorbitan monostearate and 0.05% polyoxyethylene sorbitan monostearate.

Nelson and Winder (1961) described that the milk dried with low melting point fats have better wettability and dispersibility.

Samhammer (1966) in evaluating the properties of instant dried whole milks showed that their wettability was not greatly dependent on the amount of fat originally present. The same author (Samhammer, 1972) in another study on spray dried whole milk reported that the addition of lecithin increases wettability but itself did not produce an instant type milk. For increasing the

wettability, a mention was made by Budding (1966) for adding surface active liquid agent like 0.2-4% oleic acid, soya lecithin, olive oil etc. to the dried product.

Several methods for the determination of wettability were developed. Ashworth and Gunthardt (1954) sprinkled 2.5 g of dried milk into a disc of filter paper covering the bottom of 250 or 400 ml beaker. 17.5 ml water at 30°C was allowed to flow down the wall of the beaker and form a layer under the disc. The powder and water were left in contact for 5 minutes after which the liquid was strained through a Gooch crucible (allowing 10 seconds for emptying the beaker and 30 seconds for filtration). Finally total solids were determined in the filtrate using 5 ml aliquot. This value multiplied by a factor gave the percentage of powder wetted during 5 minutes.

Mures and House (1962) proposed a simple method for comparing the wettability of instant spray dried skims. They reported that the wetting time varied from a few seconds for good instant powders to several minutes for ordinary spray dried powders. The authors further showed that particle size was the most important factor controlling the ease of wettability.

Instant water was determined. The powder was sprinkled on the surface of water (30 ml) in a separating funnel. After a certain time (10 minutes)

2.2.1.5 Sinkability:

Hall and Hedrick (1971) defined sinkability as the rate at which dried milk sinks into quiescent water. It depends upon the size of particle, its surface and also particle density of the powder. The amount of occluded air in the particles of spray dried milk powder has a pronounced influence on sinkability and thus foam spray dried milks have very poor sinkability. The process of agglomeration improves sinkability by increasing aggregate weight and this effect is greater in non-fat dried milk than in dried whole milk.

Nelson and Winder (1961) reported that as little as 5% of the high melting point fats decreased the sinkability of milk powder. Harper et al (1963) claimed that the bulk density of dry milk for self dispersion should not be above 0.4 g/ml despite the improved sinkability.

Bullock and Winder (1958) described a method to improve the sinkability of dried whole milk and devised a simple test to estimate the percentage of milk powder sample which sank after being placed on a quiescent water surface for a given period of time, usually 30 seconds.

In the sinkability test of Bullock and Winder (1958) the rate at which the dried milk sinks into quiescent water was determined. The powder (500 mg) was sprinkled on the surface of water (50 ml at 25°C) in a separating funnel. After a certain time (20 seconds to

3 minutes), the portion of dried milk which sank was quickly drawn off together with water. After complete dissolution of this powder, total solids were determined in a 5 ml aliquot. The result was expressed as percentage sinkability.

Samhammer's (1966) photometric method for comparing the instancy of different types of powders was based on the rate of sinkability of powder particles. Neff and Morris (1967) described a new method for determining the "sinkability index" of powder where the time for all the powder to disappear from the quiescent surface was measured.

2.2.2 Chemical characteristics of dried milk:

2.2.2.1 Moisture:

Coulter et al (1951) reported that salts in solution decreased the vapour pressure by an osmotic effect. Milk fat was presumed to have no direct effect on moisture retention of dry milk. The hygroscopicity of the amorphous lactose probably has the greatest effect on the moisture equilibrium of dry milk after its manufacture.

Ashworth et al (1954) indicated that an optimum of 3.5% moisture in the dry milk gives maximum wettability. Low moisture below 2% in dry whole milk might slightly

accelerate oxidative changes compared to 3%. Further, at a moisture level below 2% the powders become less soluble while above 4% a decrease in wettability and dispersibility took place during storage.

Hall and Hedrick (1971) reported that the keeping quality of powder was related to its moisture content. Higher the moisture content of the product, lower is the keeping quality. Lactose and protein of milk solids have the greatest effect on the moisture equilibrium.

Mol (1977) observed that the moisture content of dried milk appeared to be influenced more by the viscosity of concentrate than by its total solids contents.

2.2.2.2 Fat and free fat:

Tamsma et al (1959) and Berlin et al (1964) from their studies on microscopic examination reported that the fat in dried milk occurs as globules surrounded by a membrane covering of protein nature or as free fat. In the free state, it is considered de-emulsified and no longer protected by membrane with the result that the globules are apt to flow together to form 'pools' of fat.

Roelofsen and Salome (1961) reported that the size of fat globules with the membrane in dried whole milk depends upon the efficiency of homogenization.

Reinke et al (1961) has discussed several factors for reducing the free fat in spray dried whole milk. Pisecky (1966) reviewed the incidence of free fat in dried milk. The author stated that the free fat portion increased upto 70% as the dried whole milk passed through individual handling stage.

Vilder and Martens (1974) observed that the homogenization of concentrated milk considerably decreases the free fat content of whole milk powder. Further, the powders with a low free fat content were found to exhibit a low permeability for gases.

The estimation of free fat in dry whole milk consists in extracting the fat by petroleum ether and then filtering the etherial layer followed by drying in a fat dish as in the case of Mojonnier method for fat testing (Hall and Hedrick, 1971).

Woodham and Murry (1974) stated that because of the variety of solvents and extraction conditions which might be used, care must be taken in expressing free fat contents. For example, a free fat content of 10% is excessive, if it referes to a powder basis but is acceptable if it referes to a total fat basis.

2.2.2.3 Hydroxymethyl furfural (HMF):

Hodge (1953) presented a lucid integrated scheme of some of the reactions known to play a role in Maillard type of browning.

Patton (1955) reviewed the general significance of browning reaction in milk products. The most important type of browning results from the complex reactions followed by interaction of carbonyl and amino compounds. The early stages of reactions before visible colour development were highly significant in predicting and controlling the browning process. Potter and Patton (1956) identified 5-hydroxymethyl furfural (HMF) as intermediate compound during the process of browning in evaporated milk.

Gottschalk and Partridge (1950) proposed an integrated scheme for early reaction between reducing sugars and amino compounds. Hydroxymethyl furfural (HMF) was produced via 1-amino-1,4-deoxy-2-ketoses and finally brown colour and flavour compounds were produced. The findings of Gottschalk and Partridge were supported by Hodge and Rist (1953).

Keeney and Bassatte (1959) proposed a quantitative method for determining 5-hydroxymethyl furfural by spectrophotometric measurement of the 2-thiobarbituric

acid (TBA) reaction product. Their method was suggested to be a sensitive detector of early symptoms of the browning reactions in the milk powders.

This method of Keeney and Bassette was adopted by Della Monica et al (1968) to determine HMF content in whole milk powder. Walker (1972) also used the same method and studied the flavour defects in edible casein and skim milk powder by estimating free and potential HMF in the samples. Craig et al (1960) modified the method of Keeney and Bassette and this modified method was used by Zadov (1970) to determine HMF values in ultra-heat treated skim milk from 98-145°C for 3 second.

2.3 Chocolate milk and powder:

Holland (1968) and Laglosis and Randolph (1967) stated that flavoured milk beverages have been promoted frequently as a possible means of increasing the sales of fluid milk. A variety of flavours have been tested but the only one that has achieved a note-worthy success has been chocolate. This flavour has almost a universal acceptance. In United States of America, one in every 10 glasses of milk currently consumed is flavoured milk, of which 50% is chocolate milk whereas the remaining covers a wide variety of milks including honey, fruit syrups etc.

Keeping in view the modern trend to meet the special requirement of industry and the consumer, flavoured milk has been suitably dehydrated to reduce its volume, to preserve and to readily reconstitute it in a form useful in maintaining its freshness over a considerable period. Consequently, at present a large market has developed for chocolate and other non-fat flavoured powders. When dissolved in a glass of whole milk they are said to provide the required nourishment for an instant breakfast (Webb and Whittier, 1970). Most of the information available on chocolate milk powder has been patented (Thomson and Ziemba, 1957; Peebles and Clary, 1958; Carlson et al, 1961; Shenkenberg, 1962 and Ra~~ce~~, 1971, 1972, 1973).

2.3.1. Ingredients used in the chocolate milk:

Webb and Whitter (1970) reported that the basic ingredients of chocolate milk are four in number. They are milk, cocoa, sugar and stabilizer. Sometimes, vitamin A, C, D and B-complex may also be added as well as copper, iron, calcium and phosphorus in permissible food grade forms in required quantities (Webb and Whitter, 1970).

2.3.1.1. Milk:

Holland (1968) stated that the milk used for manufacture of chocolate beverage falls into two general categories; those made from whole milk and those made from

skim or reduced fat milks. From the stand point of manufacturer, the fat-free products are easiest to manufacture because the fat separation problem is eliminated. If products are fortified with the addition of milk solids-not-fat, it is probably more desirable for both children and adults under the present day condition of fat consciousness. Skim milk, partially skimmed milk or whole milk may be used in the manufacture of chocolate milk. Reconstituted milk from low heat, non-fat dry solids, cream and water or reconstituted milk from sweetened condensed cream and water, together with non-fat dry milk solids are also used. Super-heated condensed milk should not be used for chocolate milk.

Langlois and Randolph (1967) on the basis of survey made on the 'Manufacturing practices for chocolate milk and drinks in USA' reported that 64.6% of the plants marketed chocolate milk containing over 3.25% milk fat while chocolate flavoured low-fat milk and chocolate flavoured skimmed milk were sold by 6 and 7.2% of the plants respectively.

Chocolate beverage from mixture of whey and milk has been reported by South African Dairy scientists and 3% fat milk in whey or 3:1 mixture of whey and whole milk was used. The product made from 3:1 mixture of whey and milk proved more acceptable to a taste panel than that made from 100% whey.

2.3.1.2 Cocoa/liquor chocolate:

Holland (1968) reported that the concentration of cocoa used for flavouring varies between 2.0 to 1.5% of the finished product. In the case of liquor chocolate, the concentration ranges between 1.25 and 2.25%. Cocoa and chocolate are made from the beans or seeds of cacao tree (*Theobroma cacao*) which grows on the countries near the equator. The consumption of both products is high for making beverages. They are also used in other food products like ice cream, cake and confectionery.

Processing of cocoa beans:

Cosgrove (1968) observed that the fresh beans from the cocoa pod have a strong bitter taste and must be treated to develop its flavour and colour. The seeds are first fermented to decrease the bitter taste and then dried. After drying, the cocoa beans are roasted and kernels of the beans are separated from the shells. Roasting develops flavour. The kernels are broken into small pieces called 'nibs'. These nibs are ground on stone mills which force the fibrous material to shred and cause a rupturing of the fat cells. The resulting viscous material is called chocolate liquor. This liquor contains 54% fat, known as cocoa butter. If some fat is removed from the liquor and the end solid product is pulverized then it is called as cocoa powder. It contains an average

20 to 26% fat. Some times during the initial processing of the beans, an alkali may be added to the nibs just prior to roasting. This process is known as 'dutching'. Dutching neutralizes the natural acidity present in the beans and because the action tends to soften and break up the fibres, a full release of flavour results. Commercially, chocolate is sold as Dutch or Amercian. Liquor which is not alkalized produces a powder called as 'Natural cocoa'. The difference between the natural cocoa and the Dutch processed cocoa is that the latter is less acid, less bitter, darker in colour and gives less sedimentation on storage. In general, the type of beans, fermentation, method of roasting and alkalization of bulk cocoa will largely control flavour and colour.

Constituents of cocoa:

Merory (1960) reported that cocoa contains purine, theobromine, caffeine, tannin and carbohydrates. Theobromine gives cocoa stimulating properties while tannin determines extensively the colour and taste of cocoa. Carbohydrates are present in fairly large amounts in the form of starch and sugar. However, the exact identity of the flavour substances has not been established.

Cocoa substitutes:

Kenyon et al (1980) studied the effect of cocoa substitutes on flavour and other characteristics of chocolate milk. Six commercial products are being added to whole milk at 30, 50 or even 100% replacement of cocoa. The acceptance of chocolate milk decreased as the level of substitute increased. With two exceptions, a heavy sediment resulted during storage of Experimental samples containing 50-100% substitute levels.

2.3.1.3 Sweetener:

Holland (1968) reported that the various sweetening agents used in the manufacture of chocolate milk are cane or beet sugar, corn syrup, invert sugar or honey. The sugar content generally varies between 5 and 8%. According to Langlois (1967) over 80% of the chocolate milk manufacturers in USA were using cane or beet sugar while a few were using either corn sweetener or a combination of corn and cane.

In the case of chocolate milk powder manufacture, Hall and Hedrick (1971) recommended a part of the sugar to be added in liquid mix and the remainder of sugar to be blended into the dry product. It is also possible to add total quantity of sugar before drying. The size of sugar particles for dry blending is important to

achieve a uniformity of appearance and good dispersibility characteristics of the product.

Martin (1979) suggested the economical use of high fructose maize syrups (HFMS) in chocolate milk as sweetener. The three generally recognized types of high fructose maize syrups contain 42, 55 and 90% fructose respectively. The corresponding sucrose equivalent sweetness are approximately 1.1, 1.2 and 1.5.

2.3.1.4 Stabilizer:

Holland (1968) reported that the settling of cocoa particles in chocolate milks generally occurs and it is undesirable. A stabilizer is usually added to delay or prevent settling of cocoa particles. It also helps to prevent the rising of cream. Sodium alginate or 'cocoloid Irish Moss carageenan and starch and some times milk solids-not-fat are used as stabilizers. The rate of addition varies with the stabilizer used:- 1% of starch gelatinized by heating in milk at 190⁰F, 0.3% of gelatin, addition of milk solids-not-fat as condensed skim milk, 0.2% of sodium alginate, and carageenan at a concentration of 0.04%. The presence of these substances increases the viscosity of chocolate milk. The process for stabilizing

chocolate milk using a mixture of carageenan and alginate has been patented.

Sonoeren et al (1976) showed that the use of K-carageenan of low mol. wt. as stabilizer did not prevent sedimentation whereas K-carageenan of higher mol. wt. resulted in the formation of a net work of polysaccharides and casein which prevents sedimentation. The effectiveness of stabilizer was found to be a function of its molecular weight and concentration.

2.3.2 Manufacture of chocolate milk:

Anon (1970) has described a method for producing pasteurised chocolate milk. The method involves preheating of fat standardized milk to 70-75°C, homogenization at 175-200 kg/cm², addition of 2% cocoa, 5% sugar and stabilizer, pasteurization at 75°C for 30 minutes followed by rapid cooling to 8°C prior to filling into containers.

Another method for producing chocolate milk (Holland, 1968) involves the addition of prepared syrup to the base milk followed by pasteurization. In batch method of pasteurization, temperatures of 71.1°C and above for 30 minutes are employed. In H.T.S.T. system of pasteurization, temperatures of 76.6 to 82.2°C are common and holding times vary between 15 and 25 seconds.

Souza (1980) described a method which involved the following stages: Heating at 65°C , homogenization at 180 kg/cm^2 , U.H.T. sterilization for 4 seconds at 140°C , cooling to 30°C , aseptic packaging in a tetra-pack machine directly connected to sterilizer. The addition of sodium alginate was considered essential for improving the viscosity and stability of chocolate milk. Homogenization did not adequately reduce the size of cocoa particles and at 180 kg/cm^2 pressure, it had the unwanted effect of increasing sedimentation, probably because of protein instability.

Defects in chocolate milk:

Holland (1968) reported that chocolate milk is subjected to most of defects common to fluid milk, together with some that are unique in this product. One of the chief problems in chocolate milk manufacture is creaming. Cream layer in chocolate milk will contain enough of cocoa particles to give it a darker colour than the main body of the product. Homogenization prevents or delays the ^orising of cream. Alternately, when chocolate milk is processed at a temperature of 71.1°C or above for 30 minutes, creaming is prevented. Addition of suspending agents retards creaming. Another equally important problem with chocolate milk is sedimentation. Numerous methods have been tried for the elimination of settling.

They are (i) elimination of insoluble particles (ii) increased fineness of grinding cocoa (iii) increase in the viscosity of the product, and reducing the difference in density between cocoa particles and the milk base. Fine grinding of cocoa and the use of suspending agents to increase the viscosity of milk have found the widest use.

An excessive viscosity in chocolate milk is undesirable. High viscosity gives the product a rich appearance but leaves a filmy after taste in mouth. Air entrapped is likely to be retained as bubbles in the upper part of bottle giving the product an appearance of having undergone gassy fermentation.

Thickening or gelling may be caused by (i) excessive pasteurisation temperature (ii) too much stabilizer causing over stabilization (iii) use of lime or magnesium neutralizers (iv) too high acid milk.

Product characteristics:

Hall and Hedrick (1971) reported that a good quality chocolate flavoured non-fat dry milk has a pleasing flavour blend of milk solids, sugar and cocoa. It disperses readily in hot/cold water, has minimum of cocoa settling, high density (preferably 0.5 g/ml) and good flowability in dry form.

As per Holland (1968) a good quality chocolate milk should have the following characteristics; (i) mild pleasant chocolate flavour (ii) little or no sedimentation (iii) low to medium viscosity (iv) light to medium colour (v) medium sweetness.

CHAPTER - III

SCOPE AND PLAN OF WORK

3.0 SCOPE AND PLAN OF WORK

3.1 Scope and importance of work:

There is an increasing demand in the world for instant breakfast food. This holds good in our country as well due to the rapidly improving economic conditions. This increased demand for various types of quality products is particularly noticed in upper and high middle income groups. Under these conditions Instant chocolate milk powder by virtue of its higher reconstitutability and its easy storage for a long time at room temperature provides a promising national and international market. From the preceding review of literature, it is evident that not much has been reported on the preparation of instant chocolate milk powder. Whatever little work has been done in Western countries pertains to cow milk only. But the organized dairy sector in India handles mostly buffalo milk and when fat rich products like ghee, butter etc. are produced, the skim is left behind. The milk condenseries can, therefore, divert this skim milk as well as low fat milk for the manufacture of Instant chocolate milk powder which may bring a higher profit to the ~~country~~ industry.

Keeping in view the above points, it was considered desirable to make possible improvement in the quality

characteristics of chocolate milk powder from buffalo milk using a 'single-pass' instantizing arrangement. Further, the physico-chemical characteristics of normal spray dried chocolate buffalo milk powder as affected by the total solids percentage in mix as well as instant chocolate buffalo milk powder will also be investigated.

3.2 Plan of work:

Instant chocolate milk powder will be prepared both from buffalo skim milk and buffalo double toned milk as follows:

Skim milk will be adjusted to 0.05-0.10 fat and the double toned milk to 1.5% fat. The total solids content in the chocolate milk concentrate from both skim and double toned buffalo milks will be adjusted to 35, 40 and 45%. The drying conditions will be uniformly maintained by keeping the atomizer speed at 25,000 \pm 1000 rpm and the inlet and outlet air temperatures at 190 \pm 10°C and 95 \pm 5°C respectively. The chocolate powder will be prepared by normal spray drying using all the three total solids concentrations. The concentrate yielding the best quality powder as adjudged by physico-chemical characteristics will be further utilized for the production of instant chocolate milk powder.

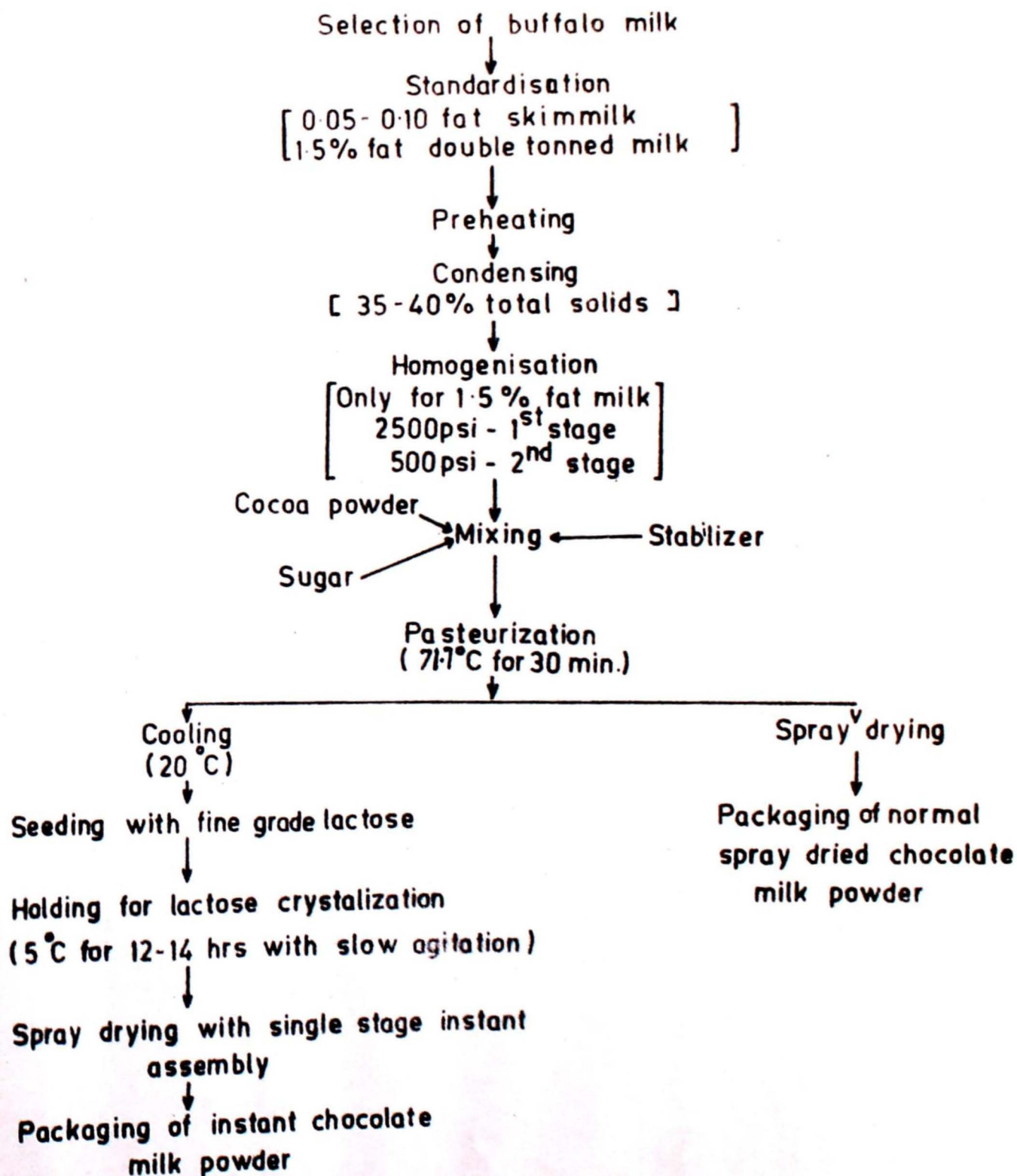
The best chosen mix concentrate will be held at 5°C for 12 to 14 hours after the addition of seeding material (i.e. fine grade lactose) with slow agitation for the optimum crystallization of lactose. The instant chocolate skim milk powder as well as instant chocolate double toned milk powder will be prepared using the same operating conditions for the drying air except the speed of atomizer which will be maintained at 10,000 , 15,000 and 20,000 rpm.

The normal spray dried as well as the Instant chocolate milk powder prepared from both buffalo skim milk and double toned milks will be examined for their physico-chemical characteristics to evaluate their quality. The characteristics studied for the purpose will include moisture, fat, free fat, pH, TBA value, wettability, sinkability, dispersibility, solubility index, average particle density, bulk density, and percent volume occupied by powder particles. However, the tests of free fat, and TBA values will be performed only on the powder samples obtained from double toned milk.

CHAPTER - IV

MATERIALS AND METHODS

FIG. 1 FLOW CHART FOR PRODUCTION OF CHOCOLATE MILK POWDER



4.0 MATERIALS AND METHODS

4.1 Production of chocolate milk powder:

Chocolate milk powder was prepared by normal spray drying method as well as by single-pass instant method. The steps involved in the manufacturing methods are depicted in Fig. 1.

1) Selection of milk:

Buffalo milk was obtained from the herd maintained at the Institute's farm. The processing of milk was carried out in the Experimental Dairy. About 500-600 litres of buffalo milk (skim or double toned) was taken and subjected to organoleptic evaluation and chemical tests viz., acidity, fat content and clot-on-boiling test.

2) Standardization:

In the case of skim milk, the fat content was maintained at 0.05 - .1% level by separation in the De-Laval Tri-process machine, whereas for double toned milk, it was standardized to 1.5% fat by mixing buffalo skim and whole milks.

3) Pre-heating:

The milk was heated in a Silkeborg plate-heater

(2500 lb per hour capacity) with steam as heating medium. A pre-heating temperature of 90°C was employed without holding.

4) Condensing:

Pre-heated milk was condensed in a vacuum pan (150 kg/hr water evaporation capacity) with steam as heating medium and using a vacuum of 26" Hg. Concentration of milk was periodically checked by drawing a sample and testing it on "Bausch and Lomb" refractometer till the desired total solids of 35-40% were reached. The steam was then stopped and the product taken out in cans.

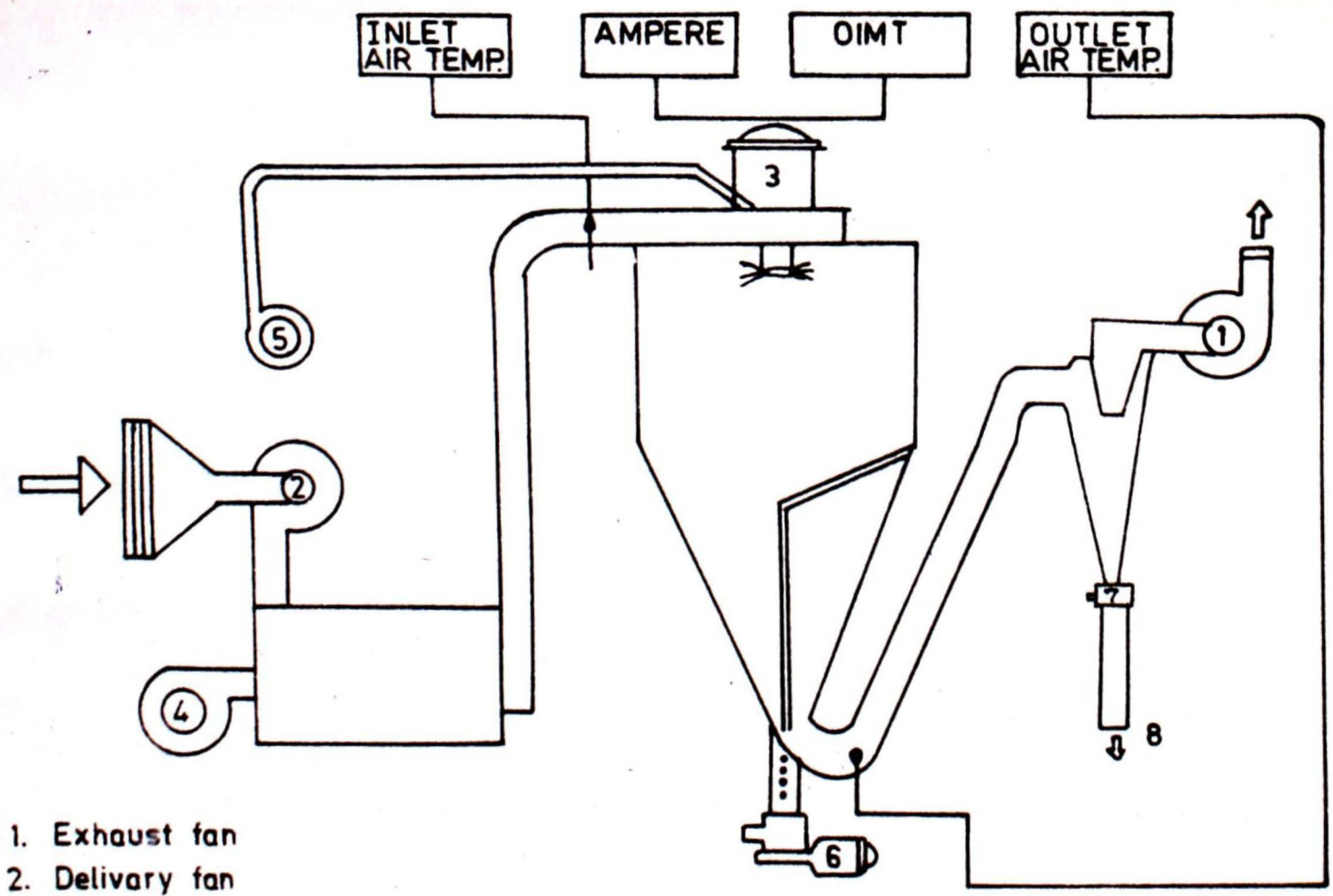
5) Homogenisation:

Homogenisation was carried out in the case of double toned milk only. A pressure of 2500 psi was used in the first stage and 500 psi in the second stage in a Gaulin double-stage homogenizer.

6) Mixing:

The calculated amounts of sugar, cocoa powder and stabilizer were added to concentrated milk and mixed. The rate of addition of above ingredients was such that the reconstituted chocolate milk will have 1.5% cocoa,

FIG.2. SPRAY DRIER FOR NORMAL CHOCOLATE MILK POWDER



- 1. Exhaust fan
- 2. Delivery fan
- 3. Atomizer
- 4. Oil burner
- 5. Feed pump
- 6. Rotating sweep
- 7. Star wheel
- 8. Chocolate powder outlet

6% sugar and 0.2% stabilizer. The stabilizer used was sodium alginate and added when mix attained a temperature of 80°C . After addition and stirring, the mix was circulated by means of a pump to get a homogenous mix. The mix was further pasteurised by batch method (71.7°C for 30 minutes) and cooled.

7) Seeding and holding:

This step is involved only for the mix which is meant for Instant chocolate milk powder manufacture. 0.2% fine grade lactose was used as seeding material. The can was kept in cold store and seeding material was added when temperature reached to about 20°C . Then the mix was slowly agitated by a stirrer and left overnight for lactose crystallization.

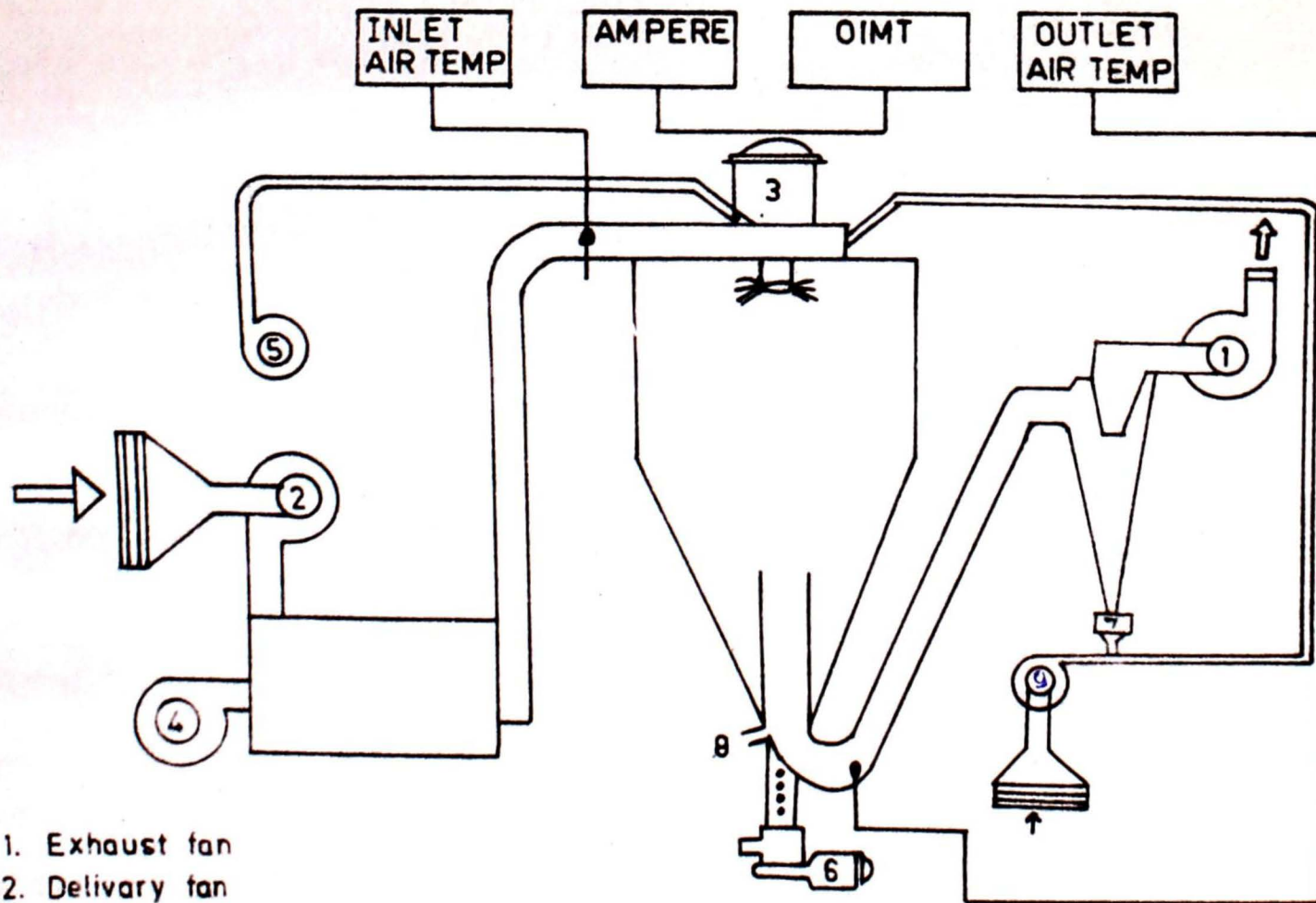
8) Drying:

The mix obtained above was dried in an Anhydro-spray drier as described below:

a) Normal chocolate milk powder:

After the plant has been assembled as in Fig. 2 and the atomizer properly placed, the motors were started in the following order: exhaust fan, delivery fan, oil burner, rotary discharge valve and scraper.

FIG. 3. SPRAY DRIER FOR INSTANT CHOCOLATE MILK POWDER



1. Exhaust fan
2. Delivery fan
3. Atomizer
4. Oil burner
5. Feed pump
6. Rotating sweep
7. Star wheel
8. Instant chocolate powder outlet
9. Air blower

When the outlet air temperature reached to about 95-100°C, the feed pump was started. The outlet air temperature was maintained at $190 \pm 10^\circ\text{C}$. The r.p.m. of the atomizer was controlled at $25,000 \pm 1,000$. The mix was divided into three portions of 45, 40 and 35% total solids concentrations and dried. Samples of powder from above total solids concentrations were collected separately and packed in polythene bags.

Replications:- Three trails with buffalo skim milk and three trails with buffalo double toned milk with the above total solids concentrations were taken.

9) Instant chocolate milk powder:

Except for the change in the discharge point of milk powder and re-circulation of the fines recovered from the escaping air in the cyclone, as shown in Fig. 3, the operating procedure followed was the same as described before. The percentage of total solids in the mix was 45 ± 0.5 and it was held at 5°C for 12-14 hours. The rpm of the atomizer was 10,000, 15,000 and 20,000 and was maintained within a range of $\pm 1,000$ during the trials. The samples obtained with the above atomizer speeds were collected and packed in polythene bags.

Replications: Three trials with buffalo skim milk and three with buffalo double toned milk for the above mentioned atomizer speeds were taken.

4.2 Analytical procedures:

4.2.1 Analysis of raw milk:

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

- i) Organoleptic tests were performed as per the procedure described in I.S. 1479 (Part I) 1960.
- ii) Fat content was determined by Gerber method using the procedure described in I.S. 1225-1958.
- iii) The acidity was determined by the method as in I.S. 1479 (Part-I) 1960.

4.2.2 Analysis of cocoa powder:

- i) Fat:- The fat content in cocoa powder was examined according to Mojonnier method as described in 'Laboratory Manual - Methods of analysis of milk and its products' 1959.
- ii) pH:- The pH of cocoa powder was determined as per the procedure given in "Laboratory Manual - Methods of analysis of milk and its products" 1959.



iii) Total ash: Total ash content of cocoa powder was determined according to the method described in IS:1164-1169.

iv) Moisture Test: - Moisture test of cocoa powder was determined as per the procedure given in IS:1164-1969.

4.2.3 Analysis of chocolate milk mix:

i) Viscosity: - Viscosity was measured by Stromer's viscometer as per the method described by Goyal (1972)-. Mixes were well mixed in a beaker for 5 minutes and the time taken for 100 revolutions of blade at 20⁰C was noted. It was expressed as seconds per 100 revolutions.

ii) Total solids:

Total solids of the mix were determined by the gravimetric Mojonnier method as per the procedure described in "Laboratory Manual-Methods of analysis of milk and its products" 1959.

4.2.4 Analysis of chocolate milk powder:

Chocolate milk powder samples were examined for their physico-chemical characteristics to evaluate their quality.

4.2.4.1 Physical characteristics of chocolate dried milk:

The samples of powder were examined for their

physical characteristic , namely solubility index, bulk density, average particle density, % vol. occupied by powder particles, wettability, dispersibility and sinkability as per the following details;

i) Solubility index:

Solubility index of chocolate milk powder was determined by A.D.M.I. method (1965). In the case of chocolate milk powder made from skim milk, 10 g were reconstituted in 100 ml of water at 23.9°C in a mixer for 90 seconds whereas in the case of chocolate milk powder made from double-tonned milk, 13 g were reconstituted in 100 ml of water. After allowing to stand for a while the foam was removed and 50 ml of reconstituted chocolate milk were centrifuged for 5 minutes in a conical graduated tube. The supernatant was decanted off and the residue was washed by dispersing it in water and centrifuging again for 5 minutes. The volume of sediment in ml obtained at the bottom of centrifuge tube was recorded and designated as solubility index.

ii) Bulk density:

Average particle density, bulk density, and percent volume occupied by powder particles were determined as follows:

Bulk density and average particle density were determined by the same test as described by Back et al (1962). A 100 ml graduated cylinder was filled with about 50 ml of hexane and stoppered. The volume of hexane (V_1) and the total weight (W_1) were recorded. Enough powder was added through a funnel to increase the volume to about 40 ml. The cylinder was placed on a level, vibration free surface. After one hour the volume of the powder (V_3), the volume of powder and hexane (V_2) and the total weight (W_2) were recorded. The volume of the floating portions of the powder were added to V_3 when making calculations. Volume were estimated to 0.5 ml and weighed to 0.05 g. Estimate was made as follows:

$$\text{Bulk density:} = \frac{W_2 - W_1}{V_3} \text{ g/ml}$$

$$\text{Average particle density} = \frac{W_2 - W_1}{V_2 - V_1} \text{ g/ml.}$$

$$\text{Percent vol. occupied by powder particles:} = \frac{V_2 - V_1}{V_3} \times 100$$

iii) Wettability:

A very convenient and suitable method developed by Muers and House (1962) for comparing the wettability of Instant spray dried powders was employed with a slight

modification. The modification was necessary due to the non-availability of satin drill specified by them (225g/sq.m having about 30 threads/cm in the warp and 20 in the weft). The fabric used in present investigation was satin drill of 221.6 g/sq. m. having about 28 thread/cm in the wrap and 19 in the weft. The second modification pertained to the dish. A tray of 21.2 x 16.3 cm (length x breadth) was used instead of the size 20 x15 cm used by them. The method is as follows;

A piece of fabric (conforming to the above specifications) measuring about 10 x 10 cm was stretched over one end of the body of a metallic can (6.5 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 as spacers to hold it in position centrally on the cloth. The tray (21.2 cm x 16.3 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 thick glass rod with sides about 8 cm long was placed in the dish and served to prevent close contact of cloth with the bottom of the dish.

With twocans 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 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 when the cloth touched the water and was stopped when the powder was completely wetted.

The temperature at which the test was carried out for skim milk chocolate powder was within the range of 20 to 23°C. For double toned chocolate milk powder the temperature was 40 ± 2°C because it should be above the melting point of fat as suggested by Pyne and Coulter (1960) and Bullock and Winder (1960).

iv) Dispersibility:

The American Dry Milk Institute adopted the dispersibility method of Stone et al (1954) for testing instant non-fat dry milk. The A.D.M.I. test adopted by Rizvi (1970) was slightly modified and is as follows:

A brass bowl subsequently called mixer (dimension 13 cm height, 13 cm top diameter and 24 cm inner diameter) fitted with an outlet of 1/2" diameter at the bottom was taken. To this was attached a rubber tube fitted with a pinch cock. The 'mixer' was clamped using a laboratory

Spectrophotometric cell. 10 mg of powder was added to

clamp and stand in such a way that it could be raised or lowered. 400 ml of water at 75°F was placed in the 'mixer' and 52 g of powder was transferred to the surface of water. The stirrer which was earlier fixed in position in the centre of the mixer was then turned on at 400 rpm and the stirring continued for 20 seconds. The pinch cock at the bottom outlet of the 'mixer' was then opened to release the contents on to a B.S. 72 mesh sieve. The fluid from the sieve was collected in a flask and diluted to 500 ml. This was then tested for total solids content. Two 10 ml portions of the diluted solution were transferred to weighed aluminium dishes and evaporated to dryness by Majonnier tester as per the method described in "Laboratory Manual - Methods of Analysis of milk and its products" 1959 for determination of total solids. The weight of solids obtained multiplied by 50 gave the dispersibility in grams.

v) Sinkability:

For measuring sinkability, Spectrophotometric method described by Samhammer (1966) and Abbot and Waite (1962) was used. The method is as follows:

3.5 ml of distilled water at 20°C was taken in Spectrophotometric cell. 10 mg of powder was then dusted

on the surface of water and percentage transmittance was measured at 760 nm in a Beckman D.U. Spectrophotometer. The readings were recorded after 2, 4 and 6 mts. after giving 6 tappings each time on the sides of the cell.

4.2.4.2 Chemical characteristics of chocolate dried milk:

The samples of powder were examined for their chemical characteristics, namely, moisture, pH, fat, free fat and TBA values as per the following details:

i) Moisture:

Moisture content of dried chocolate milk was determined gravimetrically using Majonnier method as described in "Laboratory Manual - Methods of analysis of milk and its products" 1959.

$$\text{Percentage moisture} = \frac{\text{Loss in weight}}{\text{Wt. of sample}} \times 100$$

ii) pH determination:

Ten gram of the powder sample was weighed in 25 ml beaker and made into a fine paste with a glass rod by adding 10 ml distilled water. The readings were taken in a Digital pH meter.

iii) Fat:

Fat percentage in dried chocolate milk was determined by Rose-Gottlieb method as modified by Mojonnier and described in "Laboratory Manual - Methods of analysis of milk and its products" 1959.

iv) Free fat:

For the estimation of free fat in double toned chocolate milk powder solvent extraction method as described by Hall and Hedrick (1971) was followed in the present investigation.

The results for free fat were expressed as percentage of the total fat in the sample of powder.

v) Thiobarbituric acid value:

The method of Sidwell et al (1955) was used for the determination of TBA value of chocolate milk powder. 15 g of the product was treated with 7.5 ml of HCl (3 N) and 75 ml of water added. The powder was steam distilled and 50 ml of distillate were collected in exactly 10 minutes. 20 ml of the distillate was used for TBA colour reaction. To this aliquot, 2.5 ml of TBA reagent (0.67% in glacial acetic acid) was added and the mixture heated for 35 minutes. Then the colour readings

were taken at 530 mu and results reported as absorbancy. The readings were then taken in duplicate.

4.2.5 Analysis of reconstituted chocolate milk:

i) Viscosity:

Viscosity of reconstituted chocolate milk was measured in Ostwald Viscometer according to the procedure given by Kank Bora (1981).

ii) Sediment:

Sediment of reconstituted chocolate milk was measured by filling reconstituted chocolate milk in 100 ml cylinder. The level was brought upto 100 ml mark and kept in refrigerator for overnight. The sediment at the bottom was measured in ml.

CHAPTER - V

RESULTS AND DISCUSSION

quality, the ...
which ...
physico-chemical characteristics of ...
with respect to the ...
the above sequence are presented in this chapter.

5.0 RESULTS AND DISCUSSION

For the production of a good quality chocolate instant buffalo milk powder from skim as well as from double toned milk, the optimum concentration of total solids in the mix has to be standardised. This was achieved by taking three concentrations of the mix viz. 35, 40 and 45% total solids. A concentration of 50% total solids in the concentrate was also tried, but it resulted in choking of atomizer, and proper atomization of milk particles did not take place. Therefore, the maximum concentration of total solids in the concentrate maintained during the trials was only upto 45%. The best quality normal spray dried powder obtained out of these three concentrations was further subjected to the production of instant chocolate powder, using three different speeds of the atomizer viz., 10,000, 15,000 and 20,000 rpm. Therefore, the normal spray dried product was subjected to physico-chemical evaluation to find out the best quality. The instant powder prepared from the concentrate which gave the best quality was further examined for its physico-chemical characteristics to evaluate its quality with respect to the atomizer speed. The results in the above sequence are presented in this chapter.

5.1 Standardization of the optimum concentration of mix:

Three different concentrations of mix, namely, 35, 40 and 45% total solids were prepared by blending buffalo skim or double toned milk with 1.5% cocoa powder, 6.0% sugar and 0.25% stabilizer. The fat content in skim milk was standardized to 0.10 and in double toned milk to 1.5%. The average composition of milk used in the preparation of chocolate milk concentrate mix is presented in Table-1.

Table-1

Average composition of milk used in the chocolate milk mix preparation

Sample No.	Type of milk	Organo-leptic test	C.O.B.	% acidity (L.A)	Fat %	SNF %
1.	Buffalo skim	Normal	Negative	0.159	0.10	9.75
2.	-do-	Normal	-do-	0.160	0.10	9.73
3.	-do-	Normal	-do-	0.165	0.10	9.70
	Average	-	-	0.161	0.10	9.72
4.	Buffalo double toned	Normal	Negative	0.165	1.5	9.81
5.	-do-	Normal	-do-	0.160	1.5	9.80
6.	-do-	Normal	-do-	0.165	1.5	9.81
	Average	-	-	0.163	1.5	9.80

It can be observed from the table that on an average the buffalo skim milk used in the trials contained an acidity of 0.161 as percentage lactic acid, fat percentage of 0.10 and SNF 9.73%. The average values for lactic acidity, fat and SNF in the case of buffalo double toned milk were 0.163%, 1.5% and 9.80% respectively.

The average composition of cocoa powder used in the preparation of mix is shown in Table-2.

Table-2

Average composition of cocoa powder

S.No.	Particulars	Value
1.	Moisture % by weight	3.5
2.	Fat %	14.8
3.	Ash %	4.8
4.	pH	5.4

The cocoa powder sample was kept the same for all manufacturing trials and it was analysed in duplicate to determine its moisture, fat and its ash content. The pH value of powder was also examined. The values for moisture, fat and ash were observed to be 3.5%, 14.8% and 4.8% respectively. The results indicate that this particular cocoa powder sample contained a low fat content and a low pH value and was hence a natural cocoa.

5.1.1 Viscosity of concentrated mix before spray drying:

The mix was concentrated before normal spray drying to obtain buffalo skim as well as double toned chocolate milk powder. Three concentrations of total solids viz., 35, 40 and 45% were maintained. The values on viscosity measured as seconds/100 revolutions are given in Table 3.

Table-3

Total solids concentration (%age)	Viscosity (measured as seconds/100 revolutions)	
	Buffalo skim chocolate milk	Buffalo double toned chocolate mix
35	52	49
40	78	76
45	82	80

As expected, the values of viscosity increased with the increase in total solids concentration.

5.1.2 Production of normal spray dried chocolate buffalo milk powder:

Three trials were conducted for each concentration of buffalo skim chocolate milk concentrates as well as for double toned chocolate milk concentrate. Spray drying was carried out by the normal method described under Materials and Methods (4.1).

Average physical properties of chocolate milk powder as affected by % T.S. in the concentrate.

Sr.No.	Particulars	Skim chocolate milk powder			Double toned chocolate milk powder		
		T.S. %age in concentrate			T.S. %age in concentrate		
		35%	40%	45%	35%	40%	45%
1.	Solubility Index (ml)	1.2	1.3	1.5	1.2	1.6	1.76
2.	Bulk density (g/ml)	0.638	0.665	0.74	0.632	0.66	0.704
3.	Particle density (g/ml)	1.114	1.167	1.208	1.082	1.1584	1.200
4.	Volume occupied by particles (%)	55.90	57.93	62.07	54.55	57.15	60.63
5.	Wettability (Sec.)	161.0	123.3	101.0	50.33	42.6	36.3
6.	Dispersibility (g)	33.8	38.65	41.91	32.36	34.97	39.45
7.	Sinkability (% transmittance)						
	After 2 mts.	13.7	13.5	14.8	16.9	16.9	15.3
	After 4 mts.	11.8	11.4	8.3	15.5	15.4	13.9
	After 6 mts.	11.0	10.1	9.0	13.9	14.8	12.9

5.1.3 Physical properties of normal chocolate milk powder:

The dried products obtained were analyzed for their physical characteristics viz. solubility index, bulk density, particle density, volume occupied by particles, \bar{z} , wettability, dispersibility and sinkability. The results showing the average values for three trials are presented in Table 4.

The individual values of trials have been shown in Appendix I and II. These properties have been discussed individually in relation to their statistical interpretation as under:

5.1.3.1 Solubility index:

The average values shown in Table-4 indicate that the solubility index of chocolate milk powder increases with the increase in the concentration of total solids in the mix. The increase in solubility index is comparatively more in case of double toned chocolate milk powder than skim milk chocolate powder. For example, the solubility index for skim chocolate powder from 35% Total solids concentrate increased from 1.2 ml to 1.5 ml for 45% total solid concentration, whereas it increased from 1.2 ml to 1.76 ml for the corresponding total solids concentrations for double toned chocolate milk powder.

The statistical analysis of data on solubility index has been presented in Table 5.

Table-5
ANOVA TABLE

Sources	d.f.	M.S.S.	F cal.
Among Replicates	2	0.2005	1.956
Among Types (T)	1	0.244	26.15942 **
Among Total solids (T.S.)	2	0.0238	31.9565 **
Interactions (TxT.S)	2	0.0076	3.115
Error	10		

Note: ** Significant at 5% level.

The results show that there is a significant variation in the solubility index of skim chocolate milk powder and double toned chocolate milk powder at 5% level. The same is also true for the various three concentrations of total solids in the concentrate at 5% level. Some other workers, namely, Rizvi (1970) have also reported similar results, i.e., the solubility index increases with the increase in total solids of milk concentrate. It can be concluded that solubility of skim chocolate milk powder is greater than that of double toned chocolate milk powder. Further, it can be seen in general that the solubility of chocolate milk powders is comparatively lower than the solubility of milk powders. The reason for higher solubility index of chocolate milk powders is that the cocoa particles also settled at the bottom along with heat denatured milk powder particles as a result of centrifuging and thereby depositing a slightly higher sediment in the

solubility index centrifuge tube.

5.1.3.2 Bulk density:

Bulk density of various chocolate milk powder samples has been presented in Table-4. The average values for skim chocolate powder prepared from 35, 40 and 45% concentration of total solids are respectively 0.638, 0.665 and 0.74 expressed as grams/ml. The corresponding values for chocolate milk powder prepared from double toned milk are 0.632, 0.66 and 0.704. It is evident from the values that the powder prepared from a higher degree of concentration in total solids gives rise to higher value of bulk density. This is true for both skim as well as double toned chocolate milk powder. Similar findings were reported by Rizvi (1970) who concluded that the bulk density of non-fat buffalo milk powder increased with increase in total solids concentration. Synal (1980) has also reported similar results on bulk density in his work on spray dried malted milk powder.

Further, the values of bulk density for skim milk chocolate powder are slightly greater than those obtained from double toned chocolate milk powder. Also the values of bulk density obtained in the present investigation are comparatively higher than from ordinary milk powder. This is due to the presence of added sugar in chocolate milk powder. Table 6 shows the results of statistical analysis on the data for bulk density.

Table-6.
ANOVA TABLE

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.000153	0.620
Among Types (T)	1	0.000938	3.803
Among Total solids (T.S)	2	0.124	50.440 **
Interaction (T x T.S)	2	0.0005637	2.46
Error	10		

* Significant at 5% level.

It is observed from the table that there was a highly significant difference among the three total solids concentrations.

5.1.3.3 Particle density:

The values on particle density (gram/ml) have been shown in Table-4 for skim as well as for double toned chocolate powders. It can be seen from the data that in both the cases, the particle density increases with the increase in total solids concentration in the concentrate before drying. This is due to the fact that a higher total solids concentration in the concentrate gives a higher viscosity and there by resulting in the formation of bigger particle size from atomizer, and finely a higher particle density. Further the values for particle density are slightly greater for skim chocolate powder as compared to double toned chocolate milk powder. For example, the particle density of skim and double toned chocolate milk

powders prepared from 35% total solids contents are respectively 1.114 and 1.082 g/ml. No work is available in literature on this aspect of particle density. Table 7 presents the statistical analysis of data on particle density.

Table-7
ANOVA TABLE

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.000132	0.424
Among Types (T)	1	0.00195	4.801
Among Total solids (T.S)	2	0.01517	58.3261 **
Interaction (T x T.S)	2	0.00018	0.5787
Error	10		

** Significant at 5% level

The results revealed that the effect of total solids in the concentrate on the particle density of powder is highly significant.

5.1.3.4 Volume occupied by powder particles (%age):

The data is again shown in Table 4. The volume occupied by the particles (%age) is found to increase with the increase in the total solids in the concentrate, both in the case of skim milk and double toned chocolate milk powders. Further, the values are slightly higher in the case of skim chocolate powder as compared to the values for

double toned chocolate milk powder. This is true for all the three concentrations viz. 35, 40 and 45% of total solids in the concentrate.

The statistical analysis of data on volume occupied by particles is given in Table 8.

Table-8
ANOVA TABLE

Sources	d.f	M.S.S	F cal
Among Replicates	2	0.0084	0.009
Among Types (T)	1	6.372	4.169
Among total solids (T.S)	2	57.4803	64.6656 **
Interaction (T x T.S)	2	0.1950	0.219
Error	10		

** Significant at 5% level.

It is evident from the results that the effect of total solids concentration in the concentrate on the volume occupied by powder particles is highly significant at 5% level.

5.1.3.5 Wettability:

The average values on wettability of chocolate milk powder samples have been shown in Table 4. The values for wettability (in seconds) are found to be higher for the powder prepared from a lower concentration of total solids in the concentrate as compared to that powder prepared from higher total solids concentration. This is true

both for skim as well as double toned chocolate milk powders. For example, the wettability of skim milk powder prepared from 35% total solids concentration in the concentrate was 161.1 and that prepared from 45% total solids concentration was 101.0. Similar observations has been reported by Rizvi (1970) and Ingle (1971) for their work on regular spray dried powders. Further, the data indicates that the wettability times for chocolate milk powders prepared from skim milk are very much higher than those prepared from double toned milk. This difference in wettability can evidently be due to a higher temperature used in the case of double toned chocolate powder, during the course of measurement of wettability as described under Materials and Methods(4.0). The values on statistical analysis of data on wettability are given in Table 9.

Table 9
ANOVA TABLE

Sources	d.f.	M.S.S.	F cal.
Among Replicates	2	42.055	0.607
Among Types (T)	1	32767.99	473.14 **
Among Total solids(T.S.)	2	2088.22	30.152 **
Interaction (T x T.S.)	2	818.001	11.81132 **
Error	10		

** Significant at 5% level.

The results were highly significant among the types of powder and among total solids concentration. However, the results were statistically significant among the interaction between types of powder and total solids concentration in the concentrate.

5.1.3.6 Dispersibility:

Table-4 showed the data on dispersibility (in grams) of skim as well as double toned chocolate buffalo milk powders. It is evident from the values that as the concentration of total solids in the concentrate increases, the dispersibility of the powder obtained also increases. Rizvi (1970) and Synal (1980) have also reported similar findings that the dispersibility of milk powders was found to increase with the increase in the concentration of total solids in the concentrate. In the present investigation this holds good both for skim and double toned chocolate milk powders. However, the values on dispersibility for skim chocolate powders are slightly greater than those for double toned chocolate milk powders for the respective total solid concentration in the concentrate. To illustrate the skim and double toned chocolate milk powders obtained from the concentrated containing 45% total solids possessed the values of 41.91 and 39.45 respectively for their dispersibility. Statistical

analysis of data on the dispersibility of chocolate milk powder samples is shown in Table 10.

Table-10
ANOVA TABLE

Sources	d.f.	M.S.S.	F cal
Among Replicates	2	0.1531	0.128
Among Types (T)	1	29.235	24.577 **
Among Total solids (T.S)	2	85.896	72.210 **
Interaction (T × T.S)	2	1.791	1.510
Error	10		

** Significant at 5% level

The analysis of variance on dispersibility shows that among the types of powders, there was a highly significant difference. The effect of total solids in the concentrate on the dispersibility was also statistically highly significant.

5.1.3.7 Sinkability:

The average values of sinkability for skim milk as well as double toned chocolate milk powders have been presented in Table 4. The data expressed as percentage transmittance indicate that the values for skim chocolate powder are comparatively lower than those of double toned chocolate milk powder. Further as time increases from 2 minutes to 6 ^{minutes} ~~months~~ the values keep on decreasing

in all cases of total solid concentration and the types of powder indicating thereby that the maximum opacity is obtained after 6 minutes.

Comments on the quality of powder:

Based on the above observations on the physical properties of various milk powder samples, it is concluded that the quality of powder obtained from 45% total solids concentration in concentrate is the best. This is true both in the case of skim milk as well as double toned chocolate milk powders. Hence this particular concentration of 45% total solids will be utilized for manufacture of instant chocolate milk powder samples.

5.1.4 Chemical properties of normal chocolate milk powder:

The chocolate milk powders obtained from skim and double toned buffalo milks (using different concentrations viz. 35, 40 and 45% total solids in the concentrate) were examined for their chemical properties like moisture, pH, fat, free fat and TBA value. The results indicating their statistical analysis are presented below. The average values of above chemical characteristics are given in Table 11.

Average chemical properties of chocolate powder as affected by % T.S. level in the concentrate

S.No.	Particulars	Skim chocolate powder			Double tonned ^{chocolate} conc. powder		
		Total solids 35%	%age in conc. 40%	%age in conc. 45%	Total solids 35%	%age in conc. 40%	%age in conc. 45%
1.	Moisture	3.17	3.57	3.90	3.19	3.48	3.84
2.	pH	6.90	6.91	6.91	6.90	6.91	6.90
3.	Fat	1.2	1.2	1.2	8.8	8.8	8.8
4.	Free fat	-	-	-	8.9	9.10	9.0
5.	TBA value	-	-	-	Nil	Nil	Nil.

5.1.4.1 Moisture:

The average values of moisture content in the chocolate milk powder samples are given in Table 11. The individual values have been shown in Appendix I and II.

It can be observed from data that the moisture content in the skim chocolate powder samples was greater when the percentage of total solids in the concentrate before drying was higher. For example, the moisture content in the powder prepared from 35% TS concentration was 3.17% whereas the moisture content in the powder prepared from 45% total solids concentration was 3.90. The same trend of moisture content was noticed in the case of double toned chocolate milk powder prepared from different total solids concentration in the concentrate. The reason can be that with increase in concentration of total solids, the particle size increases and hence the bigger particles are also able to retain more moisture in them during the course of drying, more so because the residence time in all cases remains the same. Rizvi (1970) has also reported similar findings in his studies on non-fat spray dried powder from buffalo milk.

The results for statistical analysis of the data on moisture content is given in Table 12.

Table 12
ANOVA TABLE

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.00051	0.002
Among Types (T)	1	0.0168	0.082
Among Total solids(T.S.)	2	0.69037	33.80 **
Interaction(T x T.S)	2	9,99127	0.006
Error	10		

** Significant at 5% level.

It is evident from the values that there is highly significant difference among the total solids concentrations

5.1.4.2. pH:

The values for pH have been presented in Table 11. It is seen from the data that the pH values were the same both for skim milk and double toned chocolate buffalo milk powders. Further, there was no difference in the pH values for various milk powder samples obtained from three different concentrations ratios (35, 40 and 45%) of total solids in the concentrate before drying. The average values of pH varied between 6.90 and 6.91. However, no information is traceable in literature on this particular aspect of study.

5.1.4.3 Fat:

The average value of fat content for skim milk chocolate powder was 1.2% whereas the value for double toned chocolate powder was 8.8% (Table 11). It can be

observed from the table that the concentration of total solids in the concentrate before drying does not made any difference in the fat content of dried product. It goes without saying that a higher value of fat content in the double toned chocolate milk powder is the outcome of a higher original fat content in the initial milk.

5.1.4.4 Free fat and TBA values:

The estimation of free fat and TBA value was carried out in the case of double toned chocolate milk powders only. The data has been presented in Table-11. The free fat content in all the powder samples (prepared from the different concentrations of total solids in the concentrate viz. 35, 40 and 45%) was almost the same and of the order of about 9% on the total fat basis. However, the measurement of TBA value indicate that no 5-hydroxy methyl furfural had been formed in all the milk powder samples. This is obvious because the powder samples are fresh and do not contain a higher fat content and thereby no flavour changes have occurred in the samples. Although many workers have estimated the TBA values of milk powders, but no work has been done on chocolate milk powders.

5.2 Production of instant spray dried chocolate buffalo milk powder:

As we have standardized the concentration of total solids in the concentrate to be 45% (described earlier), this particular concentrate was subjected to instant drying

as per the method described under Materials and Methods. The instant powders were prepared from three different speeds of the atomizer (10,000, 15,000 and 20,000 rpm) to find out as to which speed produces the best quality product. The atomizer speed of less than 10,000 rpm was also tried; but it resulted in improper atomization of concentrated milk particles and choking of atomizer owing to a higher viscosity of the concentrate.

Viscosity of the concentrate used for instant spray drying:

The concentrate mix meant for instant spray drying was seeded with fine grade lactose and was stored for 12 to 14 hour at 5°C with slow agitation for lactose crystallisation. The viscosity of the mix was measured after storage by stormers viscometer. The values of viscosity was measured as seconds/100 revolutions. The average time taken for double toned mix was 142 sec/100 revolutions and that of skim chocolate mix was 144 sec/100 revolutions. It was possible to spray dry all the above mixes.

The qualitative examination of concentrate under a microscope showed the presence of lactose crystals formed in the concentrate as result of seeding and storage at low temperature.

5.3 Physico-chemical characteristics of instant chocolate milk powders:

The chocolate milk powder samples (from both skim and double toned chocolate buffalo milk) prepared

Table 13

Average physical properties of instant chocolate powder as affected by r.p.m of atomizer

Sr. No.	Particulars	Skim chocolate instant powder			Double toned chocolate instant powder		
		Atomizer speed			Atomizer speed		
		10,000	15,000	20,000	10,000	15,000	20,000
1	Solubility Index(ml)	1.8	2.1	2.4	2.3	2.8	3.0
2.	Bulk density (g/ml)	0.63	0.62	0.59	0.60	0.58	0.54
3.	Particle density (g/ml)	1.321	1.289	1.250	1.319	1.278	1.248
4.	Volume occupied by powder particles(%)	55.09	53.41	50.20	55.11	52.13	50.94
5.	Wettability (sec)	14	17	18	4	6	7
6.	Dispersibility (g)	49.21	48.26	47.7	48.81	47.62	47.37
7.	Sinkability (% transmittance)						
	After 2 mts.	7.3	8.4	9.7	7.7	9.0	9.7
	After 4 mts.	9.2	8.6	13.7	10	9.7	10.9
	After 6 mts.	11.0	9.8	13.5	9.5	11.2	14.0

above were examined for their physico-chemical characteristics to assess their quality and the results are presented below:

5.3.1 Physical properties of instant chocolate milk powders:

The chocolate milk powder samples prepared from 45% total solids concentration in the concentrate using three different speeds of atomizer (10,000, 15,000 and 20,000 rpm) were studied for their physical properties viz. solubility index, bulk density, particle density, percentage vol. occupied by powder particles, wettability, dispersibility and sinkability. The average values of the results are presented in Table 13.

5.3.1.1 Solubility index:

The data on solubility index of chocolate milk powders has been presented in Table 13 for the three different speeds of the atomizer. It is evident from the values that a higher speed of atomizer produced powder samples with a higher solubility index. This is true both for skim as well as double toned chocolate milk powders. This is due to the fact that a higher speed of atomizer produces comparatively smaller milk particles as compared to a lower speed. The removal of moisture from smaller milk particles is quicker and there by more heat damage by the drying air. This in term brings about heat

denaturation of the proteins and a lower solubility of the powder. Similar observations have been reported by Rizvi (1970) and Ingle (1971) in their studies on non-fat instant buffalo milk powder.

Table-14

Statistical analysis of data on solubility index of instant chocolate powder.

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.04666	1.1290
Among Types (T)	1	1.388	33.602 **
Among Speeds (S)	2	0.6060	14.67 **
Interaction (T x S)	2	0.0155	0.376
Error	10		

* Significant at 5% level.

Statistical analysis of data on solubility index is shown in Table 14. It is seen from the data that the difference in values was significant at 5% level among types of powder and among speeds of atomizer.

5.3.1.2 Bulk density:

The results on the average values of bulk density have been given in Table 13. The data reveals that the bulk density of chocolate milk powder samples decreases with the increase in speed of atomizer. This is true in both the types of powder viz. skim milk and double

tonned chocolate milk powder. Further, the values for skim milk powders are comparatively slightly higher than those for double tonned milk powder. This is obviously due to the difference in their fat contents. For example, for a speed of 20,000 rpm of the atomizer, the values of bulk density for skim and double tonned chocolate milk powders were respectively 0.59 and 0.54 expressed as g/ml. Rizvi (1970) and Ingle (1971) in their work on instant buffalo skim milk powders have also reported such differences which resemble our findings.

Table 15-

Statistical analysis of data on bulk density of instant chocolate powder.

Sources	d.f.	M.S.S.	F cal.
Among Replicates	2	0.000038	0.66
Among Types (T)	1	0.008455	143.49 **
Among Speed of Atomizer (S)	2	0.00335	56.98 **
Interaction (T x S)	2	0.0000666	1.13207
Error	10		

** Significant at 5% level.

The analysis of variance (Table 15) gives the statistical interpretation of data on bulk density. It can be seen that there is a significant difference at 5% level among the types of powder and among the speeds of atomizer.

5.3.1.3 Particle density:

Table-13 shows the observations recorded on particle density of chocolate milk powders. It can be seen from the average values in the table that the particle density of powder decreases with the increase in speed of atomizer. This holds good in both skim as well as double toned chocolate milk powders. Further, the corresponding values for particle density of skim milk powder are very slightly higher than those for double toned chocolate milk powder. Although, no worker has worked on the chocolate milk powder, but Rizvi (1970) and Ingle (1971) in their work on instant non-fat buffalo milk powder have reported similar observations.

Table-16

Statistical analysis of data on particle density of instant chocolate powder.

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.000184	2.52
Among Type (T)	1	0.000102	1.40
Among Atomizer r.p.m. (S)	2	0.00711	97.12 **
Interaction (T x S)	2	0.0000120	0.16
Error	10		

** Significant at 5% level.

Statistical analysis of the data on particle density is given in Table-16. The results reveal that among the atomizer speeds, there is significant difference at 5% level.

5.3.1.4 Percent volume occupied by powder particles:

The average values have been reported in Table-13. It can be seen from the data that the percent volume occupied by the powder particle shows some decrease with the increase in the speed of atomizer. For example, in the case of skim milk powder the value decreases from 55.09 to 50.20 when the speed of atomizer is increased from 10,000 to 20,000 rpm. A similar trend in the values is observed for double toned chocolate milk powders. However, there is almost no difference between the values of skim and double toned milk powders. Rizvi (1970) and Ingle (1971) have reported similar findings during their investigation on instant skim milk powders.

Table 17

Statistical analysis of data on volume occupied by powder particles of instant chocolate powder,

Sources	d.f	M.S.S	F cal.
Among Replicates	2	14.28	1.437
Among Types (T)	1	7.38	0.743
Among rpm of atomizer (S)	2	40.87	4.113 *
Interaction (T x S)	2	3.98	0.401
Error	10		

* Significant at 1% level.

The analysis of variance table 17 presents the statistical interpretation of the data on the volume occupied by powder particles. It is clear from the results that the ~~difference~~ difference is significant (at 1% level) only among the atomizer speeds.

5.3.1.5 Wettability:

The observations on wettability have been shown in Table 13. It can be seen from the average values that wettability time increases with the increase in atomizer speed. This holds good in both the types of powders viz. skim and double toned. However, the values for wettability times are comparatively much higher for skim chocolate milk powders as compared to those double toned chocolate milk powders. This is due to fact that measurement for skim milk powders have been made at 20°C whereas for double toned chocolate milk powders at 40°C. The wettability time naturally goes down at an elevated temperatures. Rizvi (1970) and Ingle (1971) have reported comparatively higher values of wettability times than our values in the present investigation. The difference can be attributed to the compositional difference in the powders and more so presence of sugar in our product. Of course their work was on instant skim buffalo milk powders.

Statistical analysis of the data on wettability of powder samples is given in Table 18. It is observed

Table 18

Statistical analysis of data on wettability
of instant chocolate powders

Sources	d.f.	M.S.S.	F cal.
Among Replicates	2	1.555	2.058
Among Types (T)	1	533.55	706.17 **
Among Atomizer(S) rpm	2	15.388	20.36 **
Interaction(T x S)	2	2.05	2.720

** Significant at 5% level.

that there is significant difference (at 5% level) in wetting times of the two types of powders and among the atomizer speeds.

5.3.1.6 Dispersibility:

The average values on dispersibility expressed in grams have been shown in Table 13. It can be observed from the data that the dispersibility values decrease slightly with the increase in atomizer speed, both in the case of skim as well as double toned chocolate buffalo milk powders. For example, in the case of skim milk powder, the value decreases from 49.21 to 47.70 when two atomizer speed was increased from 10,000 to 20,000 rpm. Between the types of powders, it is observed that the dispersibility values were comparatively slightly greater in the case of skim milk powder as compared to double toned milk powder.

Rizvi (1970) and Ingle (1971) have also reported differences on dispersibility values of instant powders as a result of change in atomizer speed. Their findings resemble our observations on instant chocolate milk powders.

Table-19

Statistical analysis of data on dispersibility of instant chocolate powders

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.05711	0.14879
Among types (T)	1	0.9888	2.5762
Among R.P.M. of (S) Atomizer	2	8.862	8.323 **
Interaction	2	0.0162	4.330
Error	10		

** Significant at 5% level.

Statistical analysis of data on dispersibility of powder is given in Table 19. The results indicate that there is significant difference in dispersibility at 5% level among the different speeds of atomizer.

5.3.1.7 Sinkability:

The data shown in Table 13 has presented the average values for sinkability of the powder samples. It is evident from the table that the values increases with increase in measurement times in all the three speed of atomizers. This is true in both the types of powders, i.e.

skim as well as double toned powders. Further, the values are found to be increased when the speed of atomizer is increased. Also it is observed that the values are comparatively lower in the case of skim milk powder as compared to those for double toned powders. Rizvi (1970) and Ingle (1971) in their studies on non-fat instant buffalo milk powders have reported similar trend of values. However, their values were somewhat higher than our values and this can be attributed to the compositional differences of the powders.

Comments on quality of powder:

The foregoing information on physical characteristics of instant chocolate buffalo milk powders indicates that good quality skim as well as double toned chocolate milk powders can be prepared conforming to the desired A.D.M.I specifications for instant powders with respect to dispersibility. Further it can be concluded from the results, that a lower speed of 10,000 rpm of the atomizer produces a superior quality of powder possessing a higher solubility, dispersibility and wettability characteristics.

5.3.2 Chemical characteristics of instant chocolate powder:

Skim and double toned instant chocolate milk powders were examined for their moisture, pH, fat, free fat and T.B.A. values. The results are presented in Table-20.

Table 20

Average chemical properties of instant chocolate powder as affected by speed of atomizer.

Sr. No.	Particulars	Skim instant powder			Double toned instant powder		
		Speed of atomizer in rpm			Speed of atomizer in rpm		
		10,000	15,000	20,000	10,000	15,000	20,000
1.	Moisture	4.67	4.45	4.1	4.6	4.15	3.86
2.	pH	6.9	6.9	6.9	6.9	6.9	6.9
3.	Fat	1.2	1.2	1.2	8.8	8.8	8.8
4.	Free fat	-	-	-	10.1	10.8	11.0
5.	TBA value	-	-	-	Nil	Nil	Nil.

5.3.2.1 Moisture content;

The average values of moisture content in skim and double toned instant powder samples are presented in Table 20. The results indicate indicate that the powder samples obtained with a higher speed of atomizer contained comparatively lesser moisture content. The trend is similar in both the types of powder. For example, skim chocolate powder prepared with 20,000 and 10,000 atomizer speeds possessed a moisture content 4.1 and 4.67 respectively on an average. The reason for a lower moisture content for a higher atomizer speed is due to the fact that the milk particles will be smaller in size and a better drying will result. Since the residence time is kept constant therefore, the moisture content in bigger milk particles (from low atomizer speed of 10,000 rpm) will be comparatively more because of lesser efficiency in drying. Similar findings have been reported by Rizvi (1970) and Ingle (1971) in their studies on instant buffalo milk powder.

Table-21

Statistical analysis of data on moisture content of instant chocolate powders

Sources	d.f	M.S.S.	F cal.
Among Replicates	2	0.0062	0.33166
Among Types (T)	1	0.2520	13.4839 **
Among r.p.m. Atomizer (S)	2	0.563	30.139 **
Interaction (T x S)	2	0.0292	1.544
Error	10		

** Significant at 5% level.

Statistical analysis of the data on moisture content is shown in Table-21. It can be observed that the effect of types of powder and the atomizer speed is highly significant on the moisture content of the powder samples.

5.3.2.2 pH:

The average values of pH for the various milk powder samples are given in Table-20. The results indicate that there is no difference in the pH value of skim and double toned instant chocolate powders and also the atomizer speed has no effect on the pH value of powder samples. On an average, the pH value for the samples was observed to be 6.9. No information is available in literature on pH value of instant chocolate buffalo milk powders.

5.3.2.3 Fat, free fat and TBA values:

The results on fat and free fat contents of instant chocolate powders are shown in Table 20. It can be seen from the data that the fat content in skim and double toned chocolate powders was 1.2 and 8.8 respectively on an average. Further, the atomizer speed does not make any difference in the fat content of the product obtained.

Free-fat content was examined in the case of double toned chocolate powder only. The values indicate

that the amount of free-fat in the powder increases slightly with an increase in atomizer speed. To illustrate, the free fat content in the powder samples prepared with atomizer speeds of 10,000 and 20,000 was respectively 10.1 and 11.0

TBA values were estimated in the fresh double tonned chocolate milk powder samples. No measureable amount of 5-hydroxy-methyl furfural formed in the samples could be observed.

5.4 Quality of chocolate milk powder after reconstitution:

The best quality normal spray dried chocolate milk powders as well as the best quality instant spray dried chocolate milk powders were evaluated for their viscosity and sediment contents after reconstitution. The reason being that these two properties are a measurement of the quality of reconstituted product.

Table 22

Viscosity and sediment of reconstituted chocolate milk from normal spray dried chocolate powder and instant chocolate powder

Types of powder	Viscosity <i>10 Cp</i>	Sediment <i>(ml)</i>
1. Normal spray dried chocolate powder		
a) Skim	10.55	7.0
b) Double tonned	10.69	7.2
2. Instant chocolate powder		
a) Skim	10.59	10.2
b) Double tonned	10.91	10.6

5.4.1 Viscosity of reconstituted chocolate milk:

It is observed from the values reported in Table-22 that there is almost no difference in the viscosity of reconstituted milks prepared from skim and double toned instant chocolate milk powder. Further, no differences were observed in the values of viscosity for the reconstituted milk prepared from regular spray dried and instant spray dried powders. The viscosity values for skim milk powders obtained by regular and instant spray drying methods were respectively 10.55 and 10.59 expressed in centipoise.

5.4.2 Sediment in reconstituted chocolate milk:

The average values for sediment obtained in chocolate reconstituted milk samples, after a storage of 12 to 14 hr in a refrigerator have been shown in Table 22. It is clear from the results that instant reconstituted chocolate milk deposited more sediment as compared to regular spray dried reconstituted chocolate milk. For example, the reconstituted skim milks prepared from instant dried and regular spray dried methods possessed the sediment values (in ml) of 10.4 and 7.0 respectively.

A higher sediment from instant powders can be attributed to a higher heat damage caused in single-pass instantization as compared to normal spray drying. The higher heat damage is the result of more time taken during the drying process in the case of instantization method, although the temperature of drying is the same.

CHAPTER - VI

6.1 The samples of milk powders prepared from buffalo milk and cow milk were subjected to various tests for the purpose of quality evaluation. The results of these tests are given in the following tables.

6.2 The samples of milk powders prepared from buffalo milk and cow milk were subjected to various tests for the purpose of quality evaluation. The results of these tests are given in the following tables.

6.3 Buffalo milk samples were obtained from the Institute to produce normal spray dried milk and buffalo double toned milk. The concentration of milk was carried out in a vacuum pan and the stabilizer were 0.5, 0.1 and 0.2 percent, respectively. The final reconstituted chocolate milk.

6.4 The concentration of milk was carried out in a vacuum pan and the stabilizer were 0.5, 0.1 and 0.2 percent, respectively. The final reconstituted chocolate milk.

6.5 The samples of milk powders produced above were examined for their quality evaluation with respect

SUMMARY AND CONCLUSIONS

- 6.1 Significance of the present investigation and its commercial importance have been brought out under the chapter of 'Introduction' (1.0)
- 6.2 A critical review of literature with special reference to instantization methods and physico-chemical characteristics of instant milk powders has been made under Chapter-2.
- 6.3 Buffalo milk samples were obtained from the herd maintained at the Institute's Farm. Processing of milk was carried out at the Experimental Dairy of the Institute to prepare normal spray dried as well as instant spray dried powders using both buffalo skim milk and buffalo double toned milk.
- 6.4 The concentration of milk was carried in the vacuum pan and the additions of cocoa powder, sugar, and stabilizer were made to achieve their 1.5, 6.0 and 0.2 percent concentrations respectively in the final reconstituted chocolate milk.
- 6.5 The samples of milk powders produced above were examined for their quality evaluation with respect

to physical properties like solubility index, bulk density, particle density, volume occupied powder particles, wettability, dispersibility and sinkability. The chemical properties studied included moisture content, pH, fat, free fat, and TBA values. The methods used for the estimation have been described under the Chapter 4.0

6.6 The best concentration of total solids in the concentrates mix before drying was selected on the basis of the quality of normal spray dried chocolate milk powder. For this purpose, three concentration of total solid, viz. 35, 40 and 45% were tried. It was observed that a concentration of 45% TS in the concentrate yielded the best results.

6.7 For the production of instant spray dried chocolate milk powders, the concentrated mix containing 45% total solids was seeded with fine grade lactose, held for 12 to 14 hours at 5°C and then subjected to instant drying using three different speeds (10,000, 15,000 and 20,000 rpm) of the atomizer in a single-pass instant assembly.

6.8 Atomizer speed of 10,000 rpm was found to produce the best quality instant powder both from skim

as well as double toned chocolate buffalo milks. The quality of the dried product was adjudged on the basis of physical characteristics with special emphasis on solubility, including wettability, dispersibility and sinkability.

6.9 The samples prepared in the present investigation were observed to conform to the A.O.M.I. specifications on instant grade powders pertaining to their dispersibility and moisture content.

6.10 In the light of above, it is concluded that a good quality instant chocolate milk powder can be prepared from buffalo skim milk and buffalo double toned milk using a single-pass instantization method.

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Details of material and its use
 and concentration in the

Characteristics	45%	70%
1. Moisture (%)	1.24	1.24
2. pH	6.10	6.10
3. Free Ca	1.24	1.24
4. Solubility (in %)	1.24	1.24
5. Positive density	1.24	1.24
6. Bulk density	1.24	1.24
7. Vol. weight	1.24	1.24
8. Stability	1.24	1.24
9. Storage	1.24	1.24
10. Other	1.24	1.24

APPENDIX

Appendix I

Details of effect of variation in percent total solids in skim concentrate on quality of normal powder

Characteristics	35%	40%	45%	35%	40%	45%	35%	40%	45%
	I			II			III		
1. Moisture (%)	3.24	3.521	4.1	3.3	3.7	3.9	3.1	3.5	3.7
2. pH	6.9	6.9	6.9	6.9	6.9	6.9	6.8	6.8	6.8
3. Fat %age	1.21	1.21	1.28	1.41	1.48	1.39	1.20	1.9	1.20
4. Solubility Index(ml)	1.1	1.3	1.5	1.2	1.3	1.5	1.3	1.4	1.5
5. Particle density (g/ml)	1.1132	1.1847	1.225	1.128	1.145	1.1921	1.101	1.1732	1.207
6. Bulk density (g/ml)	0.628	0.684	0.732	0.631	0.661	0.758	0.6121	0.652	0.7321
7. % Vol. occupied	55.281	57.911	62.52	57.12	58.21	61.21	55.31	57.62	61.50
8. Wettability	2 mt. 32 sec.	1 mt. 58 sec.	1 mt. 31 sec.	2 mt. 52 sec.	2 mt. 12 sec.	1 mt. 40 sec.	2 mt. 39 sec.	2 mt. 0 sec.	1 mt. 52 sec.
9. Sinkability (% transmittance)									
After 2 mts.	14.0	13.8	12.5	13.7	13.5	14.8	14.1	13.9	11.9
After 4 mts.	12.1	11.5	8.2	11.8	11.4	8.4	11.8	11.7	8.3
After 6 mts.	11.3	10.8	8.3	11.0	13.1	9.0	11.1	10.1	8.2
10. Dispersibility (g)	34.21	38.72	42.12	33.21	37.51	41.21	34.21	39.21	92.20

Appendix II

Details of effect of variation in percent total solids in
double toned concentrates on quality of normal powder.

Characteristics	35%	40%	45%	35%	40%	45%	35%	40%	45%
	I			II			III		
1. Moisture (%)	3.12	3.41	3.81	3.20	3.35	3.79	3.21	3.68	3.94
2. pH	6.90	6.91	6.91	6.82	6.83	6.82	6.90	6.90	6.90
3. Fat %age	8.63	8.64	8.61	9.12	9.21	9.01	8.14	8.64	8.82
4. Free fat %	8.129	8.923	9.237	7.94	8.421	8.671	8.032	8.521	9.421
5. Solubility Index(ml)	1.2	1.5	1.8	1.4	1.6	1.7	1.2	1.8	1.8
6. Particle density (g/ml)	1.0711	1.154	1.201	1.1081	1.1451	1.1741	1.0682	1.1721	1.184
7. Bulk density (g/ml)	0.621	0.6407	0.7069	0.631	0.654	0.6892	0.6428	0.6821	0.7222
8. % Vol. occupied	54.212	56.12	61.90	58.12	57.12	69.64	54.32	58.23	60.317
9. Wettability (sec.)	52	48	44	49	47	33	50	33	32
10. Sinkability (% transmittance)									
After 2 mts.	17.8	16.8	15.5	18.0	17.1	15.3	16.9	16.8	15.0
After 4 mts.	15.2	15.1	14.1	16.4	15.5	15.2	15.0	15.5	12.8
After 6 mts.	13.0	15.2	12.8	15.2	15.1	11.9	14.3	13.0	12.9
11. Dispersibility (g)	33.81	35.21	38.22	32.1	35.41	41.0	31.18	34.18	39.14

Appendix III

Details of variation in atomizer speed on quality of instant skim powder

Characteristics	Speed →	10,000	15,000	20,000	10,000	15,000	20,000	10,000	15,000	20,000
			<u>I</u>		<u>II</u>			<u>III</u>		
1. Moisture (%)		4.82	4.46	4.27	4.62	4.57	4.3	4.59	4.32	4.01
2. pH		6.9	6.9	6.9	6.9	6.9	6.9	6.8	6.8	6.8
3. Fat %age		1.21	1.21	1.21	1.41	1.41	1.41	1.21	1.90	1.90
4. Solubility Index(ml)		1.8	2.0	2.6	2.0	2.4	2.6	1.8	2.0	2.2
5. Particle density (g/ml)		1.302	1.276	1.247	1.342	1.291	1.252	1.321	1.283	1.251
6. Bulk density (g/ml)		0.630	0.62	0.60	0.64	0.62	0.59	0.60	0.62	0.60
7. % vol. occupied		54.21	52.10	51.30	55.23	53.21	50.14	55.14	53.91	50.41
8. Wettability (sec.)		15	18	20	14	16	19	14	16	18
9. Sinkability										
After 2 mts.		8.0	9.1	10.8	7.1	8.2	9.4	8.0	9.9	9.1
After 4 mts.		9.0	9.0	11.2	8.9	10.0	10.8	12.1	10.1	10.8
After 6 mts.		7.2	11.0	14.0	10.3	9.4	14.0	11.0	13.4	14.2
10. Dispersibility (g)		48.40	47.37	46.91	48.82	47.31	47.12	49.21	48.20	48.91

Appendix IV

Details of variation in atomizer speed on quality of double toned instant powder.

Characteristics	10,000	15,000	20,000	10,000	15,000	20,000	10,000	15,000	20,000
	<u>I</u>			<u>II</u>			<u>III</u>		
1. Moisture (%)	4.18	4.21	3.90	4.59	4.12	3.81	4.80	4.12	3.87
2. pH	6.90	6.90	6.90	6.82	6.82	6.82	6.91	6.91	6.91
3. Fat %age	8.63	8.64	8.64	9.14	9.12	9.13	8.74	8.64	8.71
4. Free fat (%)	10.23	11.10	10.31	9.23	10.10	11.32	10.21	11.29	11.92
5. Solubility Index (ml)	2.0	2.8	3.2	2.4	2.8	3.0	2.6	2.8	2.8
6. Av. particle density (g/ml)	1.312	1.281	1.251	1.320	1.275	1.253	1.310	1.280	1.240
7. Bulk density (g/ml)	0.61	0.59	0.55	0.60	0.58	0.54	0.59	0.57	0.55
8. % vol. occupied	54.21	52.10	51.30	55.91	52.12	50.90	55.28	52.18	51.12
9. Wettability (sec)	4	6	6	4	6	5	6	7	7
10. Sinkability									
After 2 mts.	7.1	9.0	12.8	8.0	8.1	8.2	7.0	8.2	8.1
After 4 mts.	8.0	8.2	14.1	9.5	8.7	13.2	10.2	9.0	13.8
After 6 mts.	10.8	11.3	12.9	11.4	9.0	12.8	10.8	9.1	15.0
Dispersibility (g)	49.12	48.46	48.12	49.15	48.91	48.27	49.38	47.28	48.20

VERIFIED
Plan Dept
Sd/-

