This chapter deals with the methods and materials used for standardizing the procedure for the manufacture of soft serve ice cream and the methods used for the analyses of soy-buttermilk soft serve ice cream and its ingredients. Only analytical grade reagents were used unless mentioned otherwise.

3.1 METHOD OF MANUFACTURE

To standardize the method of production of soft serve ice cream using whole soybean, buttermilk and vegetable fat, a basic general procedure outlined
below was followed. With this procedure several process variables were investigated so as to arrive at most suitable combination of the processing parameters.

3.1.1 GENERAL PROCEDURE

The general procedure included processing of soybean as a source of non-fat solids as well as fat, blending of buttermilk with soy-solids, mix preparation and preparation of soft serve ice cream.

3.1.1.1 Soybean processing.

**Soaking:** Mixed variety of food grade soybean obtained from the G.B. Pant University of Agriculture and Technology, Pantnagar (UP), was cleaned and after the removal of split, undersized, wrinkled and damaged beans was soaked in three volumes of water containing 0.5 percent sodium bicarbonate (I.R) for 12-14 hr at 25°± 5°C. The soak water was then drained off.

**Blanching:** The soaked soybeans were blanched in boiling water (containing 0.5 percent sodium bicarbonate) in a steam-jacketed stainless steel kettle for 30 min, as recommended by Nelson et al. (1976).

**Cooling and dehulling:** The blanch water was drained off and the beans cooled to room temperature with the help of tap water. The soybeans were then dehulled in a dehuller (designed and fabricated at the National Dairy Research Institute, Karnal – Fig.1) and hulls were
FIG. 1. DEHULLER
separated by floatation in water. The cotyledons were separated from the residual chalaza, plumules and other sediments with the help of a sieve. The cotyledons were held at $50 \pm 2^\circ$C if not ground immediately.

3.1.1.2 Blending of soybean with buttermilk.

The soybean cotyledons were disintegrated in a 250 litre per hour capacity micropulverizer (Frigmaires Engineers, Bombay, Fig. 2) fitted with a 15 mesh screen, along with pre-calculated and weighed amount of sweet-cream buttermilk (8.3 percent total solids and 0.5 percent fat) obtained from the Experimental Dairy of the Institute. The mixture was passed again through the micropulverizer so as to get a smooth slurry. The slurry was strained through a muslin cloth to remove coarse material such as particles of residual hulls, chalaza etc.

3.1.1.3 Soft-serve ice cream (SSI) mix preparation.

The slurry was heated to $65^\circ \pm 2^\circ$C, after addition of weighed quantities of sugar, vegetable oil and stabilizer in a steam jacketed kettle with constant stirring. Buttermilk powder (obtained from the original buttermilk by spray drying after concentrating to 35 percent solids) required to make up the shortage of buttermilk SNF, was added at this stage in the case of fresh mix to be
frozen. The mix was then filtered through muslin cloth and added with the required quantity of warm vegetable oil before homogenizing at the same temperature in a two-stage Gaulin (USA) homogenizer, by maintaining a pressure of 3000 psi in the first stage and 500 psi in the second.

The homogenized mix was then pasteurized at 85°C in a steam-jacketed kettle, and then was either cooled to 15°± 2°C for direct freezing or cooled to 60°C for subsequent spray drying.

3.1.1.4 Drying of soy-buttermilk SSI mix.

The mix was spray dried in an Anhydro (Denmark) spray dryer of 35 kg water evaporation per hour capacity (Fig.3). The outlet and inlet-air temperatures were 92°± 2°C and 190°± 2°C respectively. The speed of atomizer was controlled manually to 24,000 ± 1000 rpm. The powder was collected and temporarily packaged in 20 kg polyethylene lined Kraft paper bags.

3.1.1.5 Freezing of the mix.

Soft serve ice cream was prepared by freezing the cooled and aged (2 hr at 15°± 2°C) fresh mix or reconstituted (to 36 percent total solids) mix, using a batch freezer (Vulcan-Laval, Pune) capacity 6 litres, Fig.4). Freezing was continued until maximum
FIG. 3. ANHYDRO SPRAY DRYER
FIG. 4. VERTICAL BATCH FREEZER
overrun was obtained (usually 30 min).

3.1.2 PROCESS VARIABLES

In order to standardize the process for the manufacture of SSI, following variables were studied: (1) ratio of soy- to buttermilk-solids, (2) level of sugar, (3) source of additional fats and (4) level of stabilizer. The major criteria for the evaluation of the soft serve ice cream was the sensory score. The mix was also assessed for its dipping ability in terms of rate of dipping as well as maximum overrun that could be obtained during freezing.

3.1.2.1 Ratio of non-fat soysolids to non-fat buttermilk solids.

To optimize the proportion of non-fat soysolids to non-fat buttermilk solids in the SSI mix, five different ratios viz., 1:1, 2:1, 3:1, 4:1 and 5:1 were investigated, and judged on the basis of the sensory acceptability of the resulting product. The final non-fat solids (SNF) was maintained at a level of 12 percent in all the five types of mix.
3.1.2.2 Level of sugar.

Amongst other levels, 15 percent sugar content was found to be the most acceptable by the panel of judges. The calculated quantity of refined cane sugar was added in the mix prior to heating and homogenization. When the mix was to be spray dried, only half the required quantity of sugar was added to the mix and the remainder dry mixed after spray-drying.

Ground (200 mesh size) and sterilized (at 85°C for 2 hr) sugar was blended with the powdered mix using a dry-mixer (APV Dry Mixer - Fig.5).

3.1.2.3 Source of additional fat.

The fat content of the mix was adjusted at 8 percent. Since the total fat contributed by soybean and buttermilk amounted to only about 2 percent (for a non-fat soy solids to non-fat buttermilk solids ratio of 1:1), the remainder was added in the form of a vegetable oil. To select the most suitable oil, three different types viz., hydrogenated vegetable oil (Hindustan Lever Ltd., Bombay), coconut oil (Tata Oil Co., Bombay), and the refined groundnut oil (Ahmed Oil Mills, Bombay) were studied.

3.1.2.4 Level of stabilizer.

To see the effect of stabilizer on the quality of soy-buttermilk SSI, sodium alginate (Davars M.P.
FIG. 5. APV DRY MIXER
Food Products, Gwalior) was tried at three different levels viz., 0.1, 0.2 and 0.3 percent and as it was observed that the non-fat soysolids-to-non-fat buttermilk solids ratio of 1:1 gave most acceptable product of all the different ratios studied. Three different ratios in the vicinity of this viz., 1.1:1, 1.3:1 and 1.5:1 were used along with the different levels of stabilizer for final selection.

3.1.2.5 Addition of flavours.

In order to see the effect of artificial flavouring on the acceptability of the reconstituted SSI various synthetic flavourings were added such as strawberry, vanilla, orange and pineapple (Bush Boake Allen India Ltd., Madras).

3.1.3 Method of Manufacture of Milk-Based SSI

In order to compare the soy-buttermilk SSI, a conventional milk-based SSI was manufactured keeping its composition constant as follows: SNF - 12 percent, fat - 9 percent, sugar - 15 percent and total solids - 36 percent. The ingredients used were cream, skim milk powder (spray dried), skim milk and cane sugar. The freezer used was the same as for soy-buttermilk SSI.
into a total solid dish (previously dried and weighed) and dried in an electric oven maintained at 102°± 1°C for about 2 hr. The dish was then cooled in a desiccator and weighed. The heating and cooling was repeated at 30-min intervals till the difference in weight was less than 1 mg.

### 3.3.1.2 Protein

The method of Menefee and Overman (1940) was followed for the determination of total nitrogen of buttermilk. One ml of accurately weighed buttermilk was transferred to a 300 ml Kjeldahl flask. To it was added 2.0 g sodium sulphate, 0.14 g mercuric oxide and 5 ml sulphuric acid and digested for 45 min or till the mixture gave a clear appearance. The contents of the flask were cooled, 50 ml nitrogen-free distilled water added and the ammonia distilled with 15 ml of 50 percent sodium hydroxide (containing 0.125 percent sodium thiosulphate) into 25 ml of 5 percent boric acid solution containing 4 drops of mixed indicator (made by dissolving 100 mg methyl red and 30 mg methylene blue in 60 ml of 95 percent ethyl alcohol and then making up the volume to 100 ml with distilled water). Approximately 60 to 70 ml distillate was collected in a 100 ml conical flask. The contents of the flask were titrated with 0.02N sulphuric acid. A blank determination using distilled
water in place of sample was also carried out. The total nitrogen present in the sample was calculated as under:

\[
\text{Percent total nitrogen} = \frac{(x-y) N \times 14.007 \times 100}{w}
\]

where,

\[
x = \text{ml sulphuric acid required for sample}
\]

\[
y = \text{ml sulphuric acid required for blank}
\]

\[
w = \text{weight of sample in mg}
\]

\[
N = \text{normality of sulphuric acid used}
\]

Percent protein = percent total nitrogen \times 6.38

3.3.1.3 Fat.

The conventional Gerber fat test for milk (IS: 1224-1977) was used for this purpose, where 10 ml Gerber sulphuric acid (density, 1.4 g/ml) was transferred to the butyrometer followed by the addition of 10.75 ml of buttermilk and 1 ml of amyl alcohol. The contents were mixed by gentle shaking after stoppering and then the butyrometers were centrifuged at 1,200 rpm for 5 min. The butyrometers were immersed in water bath at 65°C ± 2°C for 5 min prior to reading the fat content on the butyrometer scale.

3.3.1.4 Ash.

Fifteen ml of buttermilk was accurately weighed and dried in a silica crucible (previously dried
and weighed) in hot air oven at 105°C. Then the sample was ignited gently on a flame and finally ashed in a muffle furnace at 550°C ± 20°C to a constant weight as per IS: 1547-1968.

3.3.1.5 Carbohydrate.

The carbohydrate content of the buttermilk was calculated by difference as under:

\[
\text{Total carbohydrate} = 100 - (\text{percent fat} + \text{percent moisture} + \text{percent protein} + \text{percent ash})
\]

3.3.2 SOYBEAN

Whole soybean and cotyledons were analysed for their total solids, protein, fat, ash and carbohydrate contents, using standard methods as described below:

3.3.2.1 Total solids.

The total solids content of soybean was determined as per 3.3.1.1 by taking 10 g of ground, well mixed soybean or cotyledons.

3.3.2.2 Protein.

The protein content of soybean or cotyledons was determined by weighing 0.1 g sample into a 300 ml kjeldahl flask and then proceeding further as in 3.3.1.2. The protein content was calculated multiplying the total nitrogen by 6.25 instead of 6.38.
3.3.2.3 **Fat.**

The fat content of soybean was determined by the method described for milk in IS: 1547-1968, with the following modifications:

One gram of well mixed powdered soybean sample was accurately weighed in a Mojonnier flask, followed by addition of 9 ml distilled water and 5 ml conc. hydrochloric acid. The contents were then heated for 15 min at 60°C while shaking constantly. This was followed by the addition of 5 ml ethyl alcohol and shaking the mixture well. Thereafter the extraction with ethers was carried out as given in the original method.

3.3.2.4 **Ash.**

The ash content of soybean was determined as in 3.3.1.4 using a 10 g sample.

3.3.2.5 **Total carbohydrate.**

This was estimated by difference as in 3.3.1.5.

3.3.3 **SOY-BUTTERMILK SSI MIX**

3.3.3.1 **Relative viscosity.**

The relative viscosity of the mix was determined with the help of Stormers Viscometer (Fig.6). Well mixed SSI mix sample was taken in a beaker and placed on the platform which was raised till the mix level
touched the mark on the shaft of the blade, then the lever was released to allow the weight to drive the blade. The time taken by the blade to make one revolution (100 divisions on the dial) in the mix (at 20°C) was recorded. Similar reading was taken for water. The relative viscosity was then calculated as follows:

Relative viscosity = \frac{\text{Time taken with mix}}{\text{Time taken with water}}

3.3.3.2 **Surface tension.**

The surface tension was determined using a Torsion balance (White Electrical Instrument Co. Ltd., England - Fig. 7), as per the procedure outlined in the instruction manual. The equipment was designed to measure the surface tension and the interfacial tension of liquids, using a platinum ring. It was pre-calibrated in Newtons (0.00 - 0.12) per meter with 240 equal divisions, and 1 Newton per meter was equivalent to 1000 dynes per cm.

The sample was taken in a watch glass and placed on the platform, which was then raised, after locking the arm carrying the ring, till the ring was immersed in the milk. The lock was then released and the platform was taken down while simultaneously moving the pointer arm to keep the index line opposite the reference line, till the ring was separated from milk. At this point the reading was recorded and calculated to report the value in dynes per cm.
FIG. 7. TORSION BALANCE
3.3.3.3 **Specific gravity.**

The specific gravity was measured by using a standard specific-gravity bottle. The mix was weighed at 20°C in the specific-gravity bottle. Similarly, weight was recorded for water under identical conditions and specific gravity calculated as under:

\[
\text{Specific gravity of mix} = \frac{\text{Weight in g of the sample}}{\text{weight in g of water}}
\]

3.3.3.4 **Whipping ability.**

While the mix was being frozen in SSI freezer a certain volume (100 ml) of the mix was drawn at 5 min intervals and weighed before putting it back into the freezer. The loss of weight of the mix due to air incorporation was recorded, overrun calculated (as in 3.3.3.5) and the latter plotted against time.

3.3.3.5 **Overrun.**

The overrun obtained in the SSI was determined by using the following formula:

\[
\text{Percent overrun} = \frac{(\text{Weight of SSI mix} - \text{weight of equal volume of SSI}) \times 100}{\text{weight of SSI}}
\]

3.3.3.6 **Melting rate.**

Immediately after freezing, the sample of known weight was placed on an iron wire mesh (8 squares per linear inch) and held undisturbed for 30 min at 30°C ± 1°C.
The molten sample was collected in a tared dish and weighed to determine the weight of melted SSI. The melting rate was calculated and expressed in percent melt as shown below:

\[
\text{Percent melt} = \frac{\text{Weight of molten SSI} \times 100}{\text{weight of SSI taken initially}}
\]

3.3.3.7 Structure of SSI.

A thin film of SSI was taken on a slide and size and number of air cells per field were determined under microscope. The slides were photographed under Leitz Large field Orthoplan microscope (Orthomat Camera, W.Germany) to show the difference between soy-buttermilk and milk-based SSI.

3.3.4 SSI MIX POWDER (SSIP)

The SSIP was examined at monthly intervals for various physico-chemical characteristics as below.

3.3.4.1 Moisture.

The moisture content of SSIP was determined as in 3.3.1.1, using 5 g sample.

3.3.4.2 Protein.

The total nitrogen was estimated by weighing 0.1 g sample and thereafter following the procedure described in 3.3.1.3. The protein content was calculated by multiplying the total nitrogen by 6.25.
3.3.4.3 Non-protein-nitrogen (NPN).

The NPN content of SSIP was estimated by the method of Baker et al. (1940), wherein 1 g sample was extracted with 40 ml of 0.8 N trichloroacetic acid for 30 min in a mechanical shaker. The suspension was centrifuged in 15 ml tubes for 10 min at 1000 rpm and the supernatent collected and pooled. Ten ml portion of this supernatent was used for nitrogen determination as in 3.3.1.3, and NPN calculated as percent of SSIP.

3.3.4.4 Fat.

The fat content was determined as in 3.3.2.3.

3.3.4.5 Free fat.

The free fat content of SSIP was determined by the method of Buma (1971). SSIP which had been dried was weighed (2.5 g) and transferred to an Erlenmeyer flask and 100 ml petroleum ether (40°C - 60°C) was added to it. This was well shaken for 30 min. The contents of the flask were filtered through a Whatman No.1 filter paper and the filtrate collected in a tared fat dish. The petroleum ether was evaporated on a Mojonnier hot plate for 15 min and then the dish transferred to an oven (at 100°C ± 1°C) for 2 hr. The dish was weighed after cooling in a desiccator. The percent free fat was calculated as under:

\[
\text{Percent free fat} = \frac{\text{Weight in g of fat} \times 100}{\text{weight in g of sample}}
\]
3.3.4.6 **Crude fibre.**

The crude fibre content was estimated by the Indian Standards method (IS: 1656-1969), wherein 2.5 g dried (to constant weight) sample was accurately weighed into a Whatman filter-paper thimble and its fat extracted for about 1 hr with petroleum ether, using Soxhlet apparatus. The fat-free material was transferred to a 1000 ml flask and to this was added 200 ml sulphuric acid (1.2 percent v/v). The contents of the flask were refluxed for 30 min, filtered (in a gooch crucible) and washed with distilled water till the residue was no longer acidic. The residue was then refluxed with 200 ml of sodium hydroxide (1.25 percent w/v) for 30 min. The residue was thoroughly washed first with distilled water (in a gooch crucible) and then with 15 ml of ethyl alcohol (95 percent). The crucible and its contents were dried at 105° ± 2°C in an oven to constant weight followed by ashing in muffle furnace at 550°C, cooled and weighed. Crude fibre content was calculated as under:

\[
\text{Crude fibre by weight} = \frac{100 \left( W_1 - W_2 \right)}{W}
\]

where,

- \( W \) = weight in g of sample taken
- \( W_1 \) = weight in g of crucible and its content before ashing
- \( W_2 \) = weight in g of crucible containing ash
Five gram sample was accurately weighed in a silica c cible and ash content estimated as in 3.3.1.4.

**Pro de value.**

The peroxide value was determined by the method of Smith (1939), wherein 5 ml glacial acetic acid was added to 5 g powder in a Mojonnier flask and warmed to 35°C for 5 min. After shaking at intervals, 45 ml chloroform was added and shaken well and then the flask centrifuged for 10 min at 80 rpm. The contents were filtered through Whatman No.1 filter paper, which was washed with 20 ml mixture of acetic acid and chloroform (1:8). Saturated potassium iodide solution (1 ml) was added to the filtrate which was diluted with 100 ml distilled water. It was then titrated with 0.01 N sodium thiosulphate solution after the addition of 1 ml starch solution (1 percent w/v) as indicator. The peroxide value was calculated as under:

\[
\text{Peroxide value as meq per 1000g fat} = \frac{(S - B) \times N \times 1000}{\text{weight in g of fat in the sample}}
\]

where,

\[
\begin{align*}
B &= \text{ml of 0.01 N sodium thiosulphate required for blank} \\
S &= \text{ml of 0.01 N sodium thiosulphate required for sample} \\
N &= \text{normality of sodium thiosulphate}
\end{align*}
\]
3.3.4.9 **Solubility index.**

The solubility index was determined by the Indian Standard (IS: 1547-1968), by dispersing 14 g sample in 100 ml distilled water at 24°C in a mixing jar. The contents were then transferred to graduated centrifuge tubes (capacity, 50 ml) and centrifuged at 150 rpm for 5 min. The supernatent was discarded and replaced with equivalent amount of distilled water and centrifuged again for 5 min at 150 rpm. Direct reading of solid material remaining at bottom was recorded as solubility index in ml.

3.3.4.10 **Bulk and average particle density and the percent volume occupied by powder particles.**

The method of Beckett *et al.* (1962) was followed for the determination of BD, APD and PVOP, wherein 50 ml hexane was poured in a 100 ml graduated cylinder and covered with aluminium foil. The volume of the hexane \( V_1 \) and the total weight \( W_1 \) were recorded. Enough powder was then added slowly through a funnel into the cylinder to increase the volume by about 40 ml. The cylinder was then covered with aluminium foil and placed at a levelled and vibration-free surface. After 1 hr the volume of the powder \( V_3 \), the volume of powder + hexane \( V_2 \) and the total weight \( W_3 \) were recorded. The volume and weight were within 0.2 ml and 0.05 g respectively.
The BD, APD and PVQP were calculated as under:

Bulk density = \( \frac{W_2 - W_1}{V_3} \)

Average particle density = \( \frac{W_3 - W_1}{V_2 - V_1} \)

Percent volume occupied by powder particles = \( \frac{(V_2 - V_1) \times 100}{V_3} \)

3.3.5 SSIP MIX AFTER RECONSTITUTION

The spray dried mix was subjected to physico-chemical and sensory analyses after reconstitution (to 36 percent total solids) before and after freezing, as described below:

3.3.5.1 Relative viscosity.

The relative viscosity was determined as in 3.3.3.1.

3.3.5.2 Surface tension.

The surface tension was determined as in 3.3.3.2.

3.3.5.3 Specific gravity.

The specific gravity was determined as in 3.3.3.3.
3.3.5.4 Overrun.
The overrun was determined as in 3.3.3.5.

3.3.5.5 Melting rate.
Melting rate was determined as in 3.3.3.6.

3.3.5.6 Sensory score.
Sensory evaluation was done as in 3.5.1.

3.4 BACTERIOLOGICAL QUALITY

The bacteriological quality of fresh mix, SSI and SSIP was carried out in terms of standard plate count and coliform test (IS: 1479 Part III - 1961). The samples for the test were prepared according to the method prescribed for ice cream mix powder in APHA (1969).

3.4.1 STANDARD PLATE COUNT

The samples were prepared and diluted in phosphate buffer. Then 1 ml of the diluted sample was transferred into a petri dish to which was later added malted tryptone dextrose agar. After mixing well it was allowed to set and then the petri dish was transferred to an incubator maintained at 37°C. The colonies were counted after 48 hr incubation.

3.4.2 COLIFORM TEST

One millilitre of diluted sample was transferred into petri dish and mixed with sterilized coliform
3.6.1 ASSUMPTIONS

In order to arrive at a realistic cost of the raw materials and the end product, certain assumptions were made. Although buttermilk is not generally sold as fluid, yet for the purpose of costing its price was taken at par with that of skim milk. Since the equipments used in this study were those which are installed in the Experimental Dairy, the capacity and cost of these only were taken into account. It was proposed that for the purpose of manufacturing about 127.8 tons of SSIP, it would require running of the equipment in 3 shifts of 8 hr each for 300 days a year. The rest of the period would be used for maintenance etc.