Physico-chemical attributes to develop formulation of ice cream mix utilizing whey concentrate preserved by hurdle technique

Abstract:

Whey is a source for a number of high-quality proteins, carbohydrates, minerals and water-soluble vitamins and presence of all these components makes it a highly nutritious product. The composition whey varies with type of whey, pH and pre-heat treatment given to whey; Whey proteins have been used for many years as highly nutritious food supplements. The biological components of whey including Lactoferrin, β-Lactoglobulin, α-Lactalbumin, glycomacropeptides and Immunoglobulin. ice cream is a frozen product obtained from cow or buffalo milk or a combination thereof or from cream, and / or other milk products, with or without addition of cane sugar, dextrose, liquid glucose and dried liquid glucose, eggs, fruits, fruit juices, preserved foods, nuts, chocolate, edible flavours and permitted food colours. So, here in this study we attribute the physico-chemical properties of ice cream mix by whey in hurdle techniques.

Keywords: Whey proteins, Physico-chemical properties, Ice cream

Introduction:

Whey solids are well known for their nutritional and functional properties and the same has been well documented (Muvihill, 1991; Mann, 1998; Jayaprakasha et al., 1999). Owing to their excellent nutritional and functional properties of whey solids find numerous applications in food and dairy industry (Jayaprakasha *et al.*, 2005; Hufmann, 1996; Mann, 1998). The discharge of whey without treatment leads to environmental pollution in terms of public health hazard. Strict and stringent regulations imposed by pollution control board and also by the environmental agencies mode scientists to look for the better ways of utilizing whey in human chain.

Whey represents about 80 to 90 per cent of original volume of milk used for preparation of cheese, paneer, chhana etc. In India the quantity of whey produced annually is estimated to be more than 3 million tonnes which accounts to about 12 lakh

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tones of valuable milk solids in terms of nutrients (Khamrui and Rajorhia, 1998; Kim et al., 1987).

An interesting possibility for the utilization of whey for human nutrition is the hydrolysis of lactose into glucose and galactose (Renner and Abd El- salam, 1991). Hydrolysis generally improves desirability (Geilman, 1993), by increased osmotic pressure, easier digestibility, increased solubility, increased sweetness and easier fermentability (Cotton, 1980, Arndt and Wehling, 1989; Timmermans, 1997).

Whey contains valuable lactose and proteins. Almost about 80 per cent of whey is wasted without being used in country. Whey carries low total solids and has very low shelf life.

Hurdle in food is defined as the substances or processing parameters or various preservation factors, inhibiting the growth of various microorganisms resulting in the death of microorganisms. Hurdle concept was first introduced Leistner (1994) and according to concept each preservation parameter is termed as hurdles. Thus, the parameters like water activity (a_w), pH, redox potential, heat treatment etc, which are bacteriostatic, bactericidal are hurdles. These hurdles may govern many preservations process and more than, one hurdle is often responsible for long shelf life of certain food products (Leistner, 1994). Whey concentrate was preserved by combined effect of lowering water activity (0.92-0.94%), low pH (5.2) and addition of potassium sorbate (0.2%), to achieve a storage life of about 3 months.

The most delightful way to consume milk and all the goodness of it is certainly through ice cream. It is not only a glamorous food but also a delicate, delicious, palatable and nutritious food. Although in recent years Ice cream and frozen desserts market in India is witnessing an explosive growth, as a result of innovations in functional ingredients, technology, distribution and communication but still the per capita consumption of ice cream in India is one of the lowest in the world, which is just 0.1 litre (Ravindra, 2004).

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Ice cream is one of the fastest growing dairy products with a growth rate of 25-35 per cent. Now a day there is great demand for low-cost ingredients which provide good functional properties. Whey concentrate is a better alternative and can certainly meet requirements. Replacement of milk solids with this by products in ice cream production not only reduce the cost of production but also improve its functional properties. Whey solids have been used to replace MSNF in variety of ice cream formulations, recommended the use of dried whey to the extent of 30-40 per cent of MSNF without affecting the organoleptic quality of ice cream. Steinsholt (1974) successfully replaced 20 MSNF with whey solids without affecting the organoleptic quality of ice cream. Lactose, if added in higher concentration causes higher sandiness to ice cream; however this defect could be reduced by hydrolyzing lactose present in whey (Arbuckle, 1986).

Therefore, it is envisaged in this project to develop formulation of ice cream mix utilizing whey concentrate preserved by hurdle technique. To standardize the process for preparation of ice cream utilizing whey preserved by hurdle technique. To study the keeping quality of whey concentrate preserved by hurdle technique.

Materials and methods:

Materials:

The following materials were used in this study for the preparation of formulated ice cream by using whey concentrate.

Whey

Fresh cheese whey was procured from SRS NDRI Adugodi Bangalore for the study. Paneer whey was procured from Students experimental dairy plant, Dairy Science College, Hebbal, Bangalore.

Enzyme

Commercially available β-galactosidase enzyme Lactozyme supplied by Novozyemes south Asia Pvt Ltd, Bangalore was used to hydrolyze lactose present in

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whey. The enzyme used was lactozyme 3000ml with an activity of 3000LAU/ml and type HG-HG (high purity in glycerol).

Citric acid

Food grade quality citric acid procured from the local market was used for preparation of paneer and for pH adjustment of paneer whey.

Whole milk

Fresh cow milk procured from Student Experimental Dairy Plant, Dairy Technology Dept at Dairy Science College, KVAFSU was used for the preparation of ice cream.

Cream

Fresh cream obtained by separating the cow milk using the cream separator was used for Ice cream mix preparation.

Skim milk powder

"Nandini" brand skim milk powder manufactured by Karnataka Milk Federation was procured from the local market to formulate ice cream mix.

Stabilizer and Emulsifier

Brand name Stamulcol ULV-1000 was used in the study procured from Lucid Colloids Ltd, Jodhpur.

Flavour

Bush brand vanilla flavour was procured from local market used for flavouring of ice cream.

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Sugar

Good quality cane sugar was purchased from the local market

Utensils and Glass wares

Stainless steel vessels of varying capacities and Stainless-steel stirrers were used at various stages of the investigation. Conical flasks, beakers, volumetric flasks, measuring jars of Borosil make were used for chemical and microbiological analysis. Glass wares were cleaned by detergents and sterilized using Hot Air Oven at 160-180°C for 2 h and used for microbiological tests.

Evaporator

Single effect, rising film vacuum evaporator of capacity 35kg water evaporation per hour (Anhydro, Copenhagen, Denmark) was used to condense whey & milk.

Analytical methods

Fat

Fat content in milk and Ice cream was determined by Gerber method as per ISI: SP 18 (Part XI) 1981.

Milk Solids Not Fat (MSNF)

MSNF content in milk was estimated by using ISI Lactometer by using the following formula

$$\textbf{SNF (\%)} = 0.25\ \textbf{CLR} + 0.2\ \textbf{F} + 0.48$$

Where, CLR-Corrected Lactometer Reading

F-Fat (content in per cent)

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Total Solids (TS)

Total solids of milk and Ice cream were determined by gravimetric method as per ISI: SP 18 (Part XI) 1981.

Protein

Nitrogen content of Ice cream was determined by Micro-Kjeldal method and protein content was computed by multiplication of nitrogen with 6.38 as per ISI: SP 18 (Part XI) 1981.

Moisture

Moisture content in dried products was determined according to the procedure given in ISI: SP 18 (Part XI) 1981.

Ash

The percentage of ash was determined by the procedure given under ISI: SP 18 (Part XI) 1981.

Lactose

Lactose content was estimated by Lane-Eynon method.

Physical attributes studies

The methods employed to analyze the physical attributes of milk, cream, and ice cream are described below.

pH

pH of the sample was measured using a digital pH meter. (Elico Pvt. Ltd.) at 25°C. About 20 ml of representative samples were used for pH determination.

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Titratable acidity

Acidity was measured as per method described in ISI: SP 18 (Part XI) 1981.

Specific gravity

Specific gravity of samples was estimated at 20°C by using a Standard specific gravity bottle of 25ml capacity by taking distilled water as a standard liquid.

Viscosity

Viscosity of samples was measured using Falling Ball Viscometer at 20°C (Gilmont Instruments, USA). The temperature of the product was maintained at 20°C in water bath by adding cold water. The measuring tube was filled with the samples and the ball was inserted through the open end of the tube. Depending upon the falling time of the ball and the expected viscosity, different balls were used. The time taken by the ball to fall through a distance of 100 mm was measured accurately using $^{1}/_{10}$ sec stopwatch. The viscosity was calculated by using the following formula.

$$\mu = K(\rho t - \rho)\, t$$

Where, **μ**= Viscosity in (cp)

t= falling time of ball (minutes)

ρt = Density of the ball (g/ml)

ρ= Density of liquid at measuring temperature (g/ml)

K= Ball constant (0.3)

Melting resistance

Melting time of ice cream was observed by weighing 10 g of sample onto a wire gauge placed on a funnel over a beaker. The time taken by the sample to completely meltdown and fall into a beaker at room temperature was recorded and expressed as melting time in min (Arbukle, 1986).

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Whipping rate

During freezing of ice cream mix at a regular interval of 2min a standard volume of mix was drawn and the overrun was calculated. The time taken by the mix to attain 90 per cent over run was computed to find out the rate of whipping.

Overrun

The overrun of ice cream was calculated on weight basis using a standard 50ml, cup by using sensitive top pan balance. Calculations were made using the following formula.

$$\% \text{ Over run} = \frac{A-B}{B} \times 100$$

Where A-weight/unit volume of Ice cream mix

B-weight/unit volume of Ice cream

Penetration value

Using a cone penetrometer (made by 'Associate Instruments Manufactures India Ltd.') penetration value was determined after hardening of ice cream. The distance in millimetre by which the cone travels in 5 sec of the sample was noted. For each sample reading were recorded at 3 different spots and the mean value was noted.

Freezing point

The freezing point of the ice cream mix was determined by using a cryoscope (Cryostar I 'Advanced Milk Instruments Manufacturer', USA).

Statistical analysis

Completely randomized design (CRD) with one way analysis of variance (ANOVA) and simple t-test were used for conducting the various experiments for the formulation of ice cream utilizing whey concentrate preserved by hurdle technique. The tests of significance were carried out as per the SPSS -10 statistical packages.

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Results and Discussion:

Physico-chemical properties of cheese whey

Cheese whey was subjected to various physico-chemical analysis and results are presented in Table -1 and 2.

Physical properties

Cheese whey had an average pH value of 5.42 with a correspondent acidity of 0.21 per cent lactic acid. The average values of specific gravity, viscosity (cP) and water activity (a_w) of cheese whey were found to be 1.024, 1.069 centi poise and 0.995 respectively. The pH of cheese whey varied between 5.39 to 5.45.

Chemical composition of cheese whey

The chemical composition of Cheese whey is presented in Table-2. The cheese whey was used in this investigation had an average total solids content of 6.68 per cent

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Table 1: Physical properties of cheese whey

Properties	Cheese whey	Range
pH	5.42	5.39-5.45
Acidity (%LA)	0.21	0.20-0.22
Specific gravity	1.024	1.021-1.027
Viscosity(cP)	1.069	1.066-1.072
Water activity(a_w)	0.995	0.992-0.998

Table 2: Chemical constituents of cheese whey

Constituents	Average %	Range %
Total solids	6.68	6.58-6.78
Protein	0.83	0.80-0.86
Fat	0.35	0.33-0.37
Lactose	4.90	4.7-5.10
Ash	0.60	0.5-0.70

which comprised of 0.83 per cent protein, 0.35 per cent fat, 4.9 per cent lactose and 0.60 per cent ash.

Process optimization for the preparation of plain whey concentrate

Plain whey concentrate was prepared by condensing of fresh whey to different levels in a falling film evaporator.

Effect of condensing whey on the physico-chemical properties of whey concentrate

Fresh cheese whey was subjected for condensing to have various total solids (40, 45, 50 and 55 per cent). The effect of level of condensing on various physical properties of condensed whey is depicted in Table-3. With the increase in concentration, the pH decreased from 4.987 to 3.84. The pH of whey was 4.987 at 16 (control) per cent total solids, whereas it was 3.97, 3.967, 3.88 and 3.84 at 40, 45, 50 and 55 per cent total solid levels. In contrast to the pH, acidity increased as the total solids level increased. At total solids 16 (control) per cent; the acidity was 0.613 per cent LA whereas at 40 per cent total solids it was 1.078 LA per cent.

The specific gravity at 16 (control) per cent total solids was 1.057 whereas at 40 per cent total solids it was 1.297. With the increase in the total solids level there was proportionate increase in the viscosity. The viscosity at 16 (control), 40, 45, 50 and 55 per cent total solids was 1.237, 17.014, 19.081, 22.94 and 26.81 cP, respectively. Similarly, with the increase in the total solids level there was a progressive decrease in the water activity of whey concentrate. The water activity (a_w) was 0.983, 0.942, 0.923, 0.901 and 0.886 at 16 (control), 40, 45, 50 and 55 per cent total solids levels.

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It is evident from Table 3 that fresh cheese whey was subjected for condensing to have various total solids (40, 45, 50 and 55 per cent). The increase in total solids content with respect to various levels of condensing was found to be significant. The protein content at 16, 40, 45, 50 and 55 per cent total solids was found to be 1.99, 4.978, 5.587, 6.213, 6.683 respectively and the result was found to be significant. The fat content at 16, 40, 45, 50 and 55 per cent TS was found to be 0.84, 2.096, 2.358, 2.62 & 2.88 respectively and the result was found to be significant.

The lactose at 16 per cent (control) solids was 11.74 whereas at 40 per cent it was 29.92 with the increase in the total solids level there was proportionate increase in the lactose. The lactose at 16 (control), 40, 45, 50 and 55 per cent total solids was 11.74, 29.92, 33.03, 36.68 and 40.34, respectively. Similarly, with the increase in the total solids level, there was progressive increase in the ash content of whey concentrate. The ash content was 1.43, 3.58, 4.04, 4.48 and 4.94 at 16 (control) 40, 45, 50 and 55 per cent total solid levels respectively.

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Table 3: Effect of condensing at different levels by using vacuum evaporator on physico-chemical properties of whey

Levels of total solids (%)	Physical attributes					Chemical Composition			
	pH	**Acidity (%LA)**	**Specific gravity**	**Viscosity (cP)**	**Water activity (a_w)**	**Protein**	**Fat**	**Lactose**	**Ash**
Control (16)	4.987	0.613	1.057	1.237	0.983	1.99	0.84	11.74	1.43
40	3.97	1.078	1.297	17.014	0.942	4.978	2.096	29.92	3.58
45	3.967	1.153	1.310	19.081	0.923	5.587	2.358	33.03	4.04
50	3.88	1.18	1.317	22.94	0.901	6.213	2.62	36.68	4.48
55	3.84	1.24	1.322	26.81	0.886	6.683	2.88	40.34	4.94
CD	0.036	0.032	0.006	0.172	0.004	0.017	0.064	0.058	0.018

*All values are average of three trials

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Effect of addition of sugar at different levels and vacuum condensing on physical properties of whey

Fresh Cheese whey added with an optimum level of sugar was subjected for condensing to best accepted level. The effect of level of condensing on various physical properties of condensed whey is depicted in Table-4. With the increase in concentration, the pH decreased from 4.987 to 3.77. The pH of whey was 4.987 at 0 (control) per cent sugar, whereas it was 3.99, 3.95, 3.89 and 3.77 at 10, 12, 14 and 16 per cent sugar levels respectively. In contrast to the pH, acidity increased as the sugar addition with condensing level increased. At sugar 0 per cent (control), the acidity was 0.613 per cent whereas at 16 per cent sugar it was 1.30 per cent.

The specific gravity at 0 (control) per cent total solids was 1.057 whereas at 16 per cent total solids it was 1.325. With the increase in the total solids level there was proportionate increase in the viscosity. The viscosity at 0 (control), 10, 12, 14 and 16 per cent sugar levels was 1.237, 17.018, 19.09, 22.33 and 27.52 cP respectively. Similarly, with the increase in the sugar level there was progressive decrease in the water activity of whey concentrate. The water activity (a_w) was 0.983, 0.925, 0.908, 0.882 and 0.879 at 0 (control), 10, 12, 14, and 16 per cent sugar levels.

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Table 4: Effect of addition of sugar at different levels and vacuum condensing on physical properties of whey

Sugar addition level (%)	Physical attributes				
	pH	Acidity (% LA)	Specific gravity	Viscosity (cP)	Water activity (a_w)
Control	4.987	0.613	1.057	1.237	0.983
10	3.99	1.076	1.312	17.018	0.925
12	3.95	1.163	1.315	19.09	0.908
14	3.89	1.191	1.321	22.33	0.882
16	3.77	1.30	1.325	27.52	0.879
CD	0.089	0.084	0.007	0.0178	0.009

*All values are average of three trials

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Effect of addition of sugar at different levels and vacuum condensing on chemical composition of whey

It is evident from Table 5 that fresh cheese whey was subjected for condensing by using best accepted level. The increase in total solids content with respect to increase in addition of sugar was found to be significant. With the increase in addition of sugar there was a progressive decrease in the protein content of whey concentrate. The protein per cent content of control, 10, 12, 14 and 16 per cent solids was 1.99, 4.976, 4.72, 4.33 and 4.15 respectively and it was found to be significant. The fat per cent content of control, 10, 12, 14 and 16 per cent solids was 0.84, 2.085, 2.056, 2.034 & 2.012 respectively and it was found to be significant.

The lactose content at control was 11.74 whereas at 16 per cent sugar level it was 35.04, with the increase in the sugar level, there was proportionate increase in the lactose. The lactose content for control 10, 12, 14, and 16 per sugar additions was 11.74, 29.92, 32.33, 33.56 and 35.04 respectively. Similarly, with the increase in addition of sugar there was a progressive decrease in the ash content of whey concentrate. The ash content was 1.43, 3.58, 3.45, 3.37 and 3.22 at control 10, 12, 14 and 16 per cent sugar levels.

Preparation of lactose hydrolysed sweet concentrated whey

Whey was subjected to lactose hydrolysis. Level of enzyme addition being one of the important processing parameters it was optimized to obtain 80 per cent lactose hydrolysis in whey. The enzyme concentration level tried to obtain 80 per cent lactose hydrolysis was optimized by keeping the temperature and pH constant as recommended by the supplier of the enzyme.

Table 5: Effect of addition of sugar at different levels and vacuum condensing on chemical composition of whey

Level of sugar addition (%)	Chemical Composition (%)			
	Protein	**Fat**	**Lactose**	**Ash**
Control (0)	1.99	0.84	11.74	1.43
10	4.976	2.085	29.92	3.58
12	4.72	2.056	32.33	3.45
14	4.33	2.034	33.56	3.37
16	4.15	2.012	35.04	3.22
CD	0.01	0.18	0.02	0.017

*All values are average of three trials

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The physical properties of whey used in this investigation are represented in Table-l. The physical properties of whey such as pH, acidity, specific gravity, Viscosity (cP) and water activity (a_w) were within the range of results reported by Jayaprakasha (1992). The whey used in this investigation had an average pH of 5.42 with the corresponding average acidity of 0.21 %LA. The specific gravity, Viscosity and water activity ($a_{w)}$ whey was found to be 1.024, 1.069 cP and 0995, respectively which were within the earlier reported values.

Table-2 represents the chemical composition of cheese whey used in this investigation. Whey had an average total solids content of 6.68 which comprised of 0.83 per cent protein, 0.35 per cent fat, 4.9 per cent lactose and 0.60 per cent ash content. The results are in agreement with the composition of whey reported by several earlier workers (Jayaprakasha *et al.,* 1994; Mandal *et al.,* 1997).

Whey concentrate was prepared by condensing whey to different levels of TS in combinations with addition of potassium sorbate followed by blending various levels of sugar. The results pertaining to these are presented in Table 3, 4 and 5 respectively.

From among the various levels of sugar tried, addition of 10.0 per cent sugar to whey was found to be optimum in combination with 0.10 per cent potassium sorbate. The levels of optimum sugar requirement helpful in providing the sweetened taste.

Fresh whey was subjected for condensing to various levels of total solids. As the level of condensing increased from the initial 16 per cent to 55 per cent, there was proportionate decrease in pH and increase in acidity which are mainly due to the concentration of the components which are responsible for pH and acidity. The increase in concentration also resulted in increase in specific gravity and viscosity of the concentrate. Similarly, as the level of concentration increased there was significant decrease in the water activity of the concentrate (Table-3) which can be mainly attributed to the constituents of soluble solutes such as added sucrose and potassium sorbate along with lactose and minerals. Water activity (a_w) of the final concentrate was 0.886. The decrease in water activity (a_w) from 0.983 to 0.886 at 50 per cent total solids level results in the increased storage stability of the condensed whey concentrate. The decrease in water activity (a_w) of the final concentrate as compared to initial whey could be attributed to the condensing as it is universally known that the dissolved constituents are the main contributors for the water activity (a_w) of any solution. Hence, there was decrease in water activity (a_w). The changes in physical properties as a

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result of concentration have been reported by several workers and our results are in agreement with the earlier workers (Jayaprakasha *et al.,* 1997). Lactose crystallization was observed when whey was condensed to 55 per cent level of total solids. Therefore, 50 per cent level of total solids in the final product was found to be optimum in this investigation.

While increasing the level of condensing from 16 to 55 per cent, there was significant increase in protein, lactose, fat and ash content of whey concentrate.

Fresh Cheese whey added with an optimum level of sugar was subjected for condensing from 16 to 55% total solids. From the initial 16 per cent to 55 per cent, there was proportionate decrease in pH and increase in acidity which was mainly due to the concentration of the components which are responsible for pH and acidity. The increase in concentration also resulted in increase in specific gravity and viscosity of the concentrate. Similarly, as the level of concentration increased there was significant decrease in the water activity of the concentrate (Table-4) which can be mainly attributed to the constituents of soluble solutes such as added sucrose and potassium sorbate along with lactose and minerals. Water activity (a_w) of the final concentrate was 0.879. The decrease in water activity (a_w) from 0.983 to 0.879 at 16 per cent added sugar in the increased storage stability of the condensed whey concentrate. The decrease in water activity (a_w) of the final concentrate as compared to initial whey concentrate could be attributed to addition of sugar and the condensing as it is universally known that the dissolved constituents are the main contributors for the water activity (a_w) of any solution. Hence, there was decrease in water activity (a_w). The changes in physical properties as a result of concentration have been reported by several workers and our results are in agreement with the earlier workers (Jayaprakasha *et al.,* 1997). Lactose crystallization was observed when whey was added with 16 per cent sugar level. Therefore, 10 per cent level of sugar addition in the final product was found to be optimum in this investigation.

Fresh Cheese whey added with an optimum level of sugar was subjected for condensing from 16 per cent to 55 per cent total solids. From the initial 16 per cent to 55 per cent, there was proportionate increase in total solids and slightly decrease in fat and protein which are mainly due to the addition of sugar which are responsible. Similarly, as the level of addition of sugar increased there was significant increase in the lactose and slightly decrease in ash content of the concentrate (Table-5).

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Conclusion

In this investigation a technology has been developed for the efficient utilization of whey for the preparation of formulated ice cream. Such formulated ice cream besides supplementing regular nutritional requirement and they provide various health benefits. The formulated ice cream not only helps in increasing the shelf life of whey but also helps in preserving whey concentrate in their natural form. Formulated ice cream besides increases the mix viscosity resulting in smoother and refreshing nature they can provide potential profit margins, which in turn add to the growth and development of dairy industry.

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