



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification ⁷ : A23G 9/00, 9/04, 9/20</p>	<p>A1</p>	<p>(11) International Publication Number: WO 00/01246 (43) International Publication Date: 13 January 2000 (13.01.00)</p>
<p>(21) International Application Number: PCT/EP99/04737 (22) International Filing Date: 5 July 1999 (05.07.99) (30) Priority Data: 98305400.8 7 July 1998 (07.07.98) EP (71) Applicant (for AE AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZA ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AE AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZA ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (71) Applicant (for IN only): HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020, Maharashtra (IN). (72) Inventors: GRAY, Sarah, Jane; Unilever Research Colworth, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). TURAN, Susan; Unilever Research Colworth, Colworth House, Sharnbrook, Bedford, MK44 1LQ (GB).</p>	<p>(74) Agent: HUGOT, Alain, Eric, Philippe; Unilever plc, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: METHOD FOR THE PREPARATION OF AN AERATED FROZEN PRODUCT (57) Abstract Using a homogeniser operating at higher pressures (ca. 2000 bar) than those conventionally used in ice cream manufacturing, it is possible to generate smaller oil droplet sizes (ca. 0.3 μm) in an ice cream premix. It allows stabilization of a larger air:water interface, leading to smaller discrete gas cells which in turn modify the organoleptic quality of the ice cream.</p>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

Method for the preparation of an aerated frozen product**Technical Field of the Invention**

5 The invention relates to a method of preparation of an
aerated frozen product such as ice cream, wherein at least
part of the aerated frozen product premix is subjected to an
ultra high pressure treatment. The invention also relates to
an aerated frozen product obtained according to this process.

10

Background to the Invention

Ultra high pressure (UHP) is a known method for killing
spores and has been suggested as a suitable route to food
15 product pasteurisation. In Japan a range of pressure de-
contaminated products such as jellies, preserves, purees and
sauces have been launched on the market (Byrne, M. (1993)
Food Engineering International, 34-38).

20 Furthermore isolated, native proteins have been subjected to
UHP. These proteins are in their native form, they have not
been treated chemically or thermally before the pressure
treatment by methods which significantly modify their protein
structure (van Camp, J; Huyghebaert, A (1995) Food Chemistry
25 54(4) 357-364; Okamoto, M; Kawamura, Y; Hayashi, R; (1990)
Agric Biol Chem 54(1) 183-189). It is generally believed that
there would be no advantage in subjecting proteins which have
already been substantially denatured by for example an
initial heat-treatment prior to UHP.

30

DE 42 26 255 discloses the treatment of cream with ultra high
pressure in order to crystallise the fat.

- 2 -

It has now been discovered that the presence of a fine microstructure is critical to produce the correct texture and quality of ice cream. Organoleptic evaluation of ice cream done by the applicant of the present invention has shown that small air cells and ice crystals are associated with increased creaminess and reduced iciness, which are recognized parameters for good quality ice cream. For example, for a given ice cream formulation, a reduction in gas cell and/or ice crystal size will enhance creamy texture (and reduce ice crystal perception, nevertheless the sensory attributes are not directly influenced by the de-emulsified fat level . However, the ice cream microstructure produced in a scraped surface heat exchanger (freezer) has been found to be unstable and both ice and air structure coarsen significantly in the time taken to harden the product to typical storage temperatures of -25°C. Therefore, an important step to achieve small gas cells in ice cream is to stabilize gas cells during hardening.

To retain the desired microstructure, it has now been found that it is necessary to generate a partial network of fat aggregates adsorbed onto the air interface to provide a steric barrier to gas cell coalescence. To generate this fat network, a proportion of the oil droplets need to partially coalesce as a consequence of the shear regime encountered within the ice cream freezer. It is known that the collision efficiency (the probability of two colliding droplets remaining permanently in contact) can be significantly affected by the initial droplet size and the protein surface coverage. The collision efficiency decreases as the droplet size decreases. However, small molecule surfactants can displace protein at the oil:water interface and allow a higher collision efficiency at a given droplet size.

- 3 -

In the processing of ice cream, an homogenization step is used to generate small oil droplets, preferably with a monomodal size distribution to allow the controlled fat destabilization under shear. For an ice cream premix, the average droplet size, $d[3,2]$, upon homogenization is typically 0.6-1.0 μm . Numerous process and product variables affect homogenization efficiency. Those which have been found to have the largest effect on the final droplet size distribution are the dispersed phase volume, the type and level of surfactant used and, in particular, the pressure applied during homogenization. It has now been found that by using an homogeniser operating at higher pressures (ca. 2000 bar) than those conventionally used (ca. 150 bar), it is possible to generate smaller oil droplet sizes (ca. 0.3 μm) in an ice cream premix.

Generation of significantly smaller, and therefore a higher number of, oil droplets can allow stabilization of a larger air:water interface, leading to smaller discrete gas cells which in turn alter the organoleptic quality of the ice cream. However, it has now been discovered that very small oil droplets will give inherently stable ice cream mixes which will not generate the desired microstructure unless the desired level of fat partial coalescence occurs. To achieve this, it is necessary to either increase the collisional force between the droplets or reduce the steric barrier to coalescence. This is achieved by either optimizing the applied shear stress during processing or by manipulating the interfacial composition by the appropriate selection of emulsifiers.

It has also been discovered that the sensory properties of ice cream is dependent on the size of the fat droplets. For

- 4 -

a given air cell size, the ice cream with the smallest fat droplets scores best on creaminess when blind tested by a trained panel.

5 Definitions

Emulsifiers

Emulsifiers are defined as in Arbuckle, W.S., Ice Cream, 4th Edition, AVI publishing, 1986, ch 6 p92-94.

10

Stabilizers

Stabilizers are defined as in Arbuckle, W.S., Ice Cream, 4th Edition, AVI Publishing, 1986, ch 6, p84- 92. They can for example be locust bean gum, carrageenan, guar gum, gelatin, carboxy methyl cellulose gum, pectin, algin products and mixtures thereof.

15

Frozen Aerated dessert

A definition of a frozen aerated dessert can be found in Arbuckle, W.S., Ice Cream, 4th Edition, AVI Publishing, 1986, ch 1, p1-3. Preferably, a frozen aerated dessert according to the invention is a milk or fruit based frozen aerated confection such as ice cream. An ice cream is a frozen food made by freezing a pasteurized mix with agitation to incorporate air. It typically contains ice, air, fat and a matrix phase and preferably;

20

25

30

- . milk/dairy fat 3 to 15 % (w/w)
- . milk solids non fat 2 to 15 % (w/w)
- . sugar and other sweeteners 0.01 to 35 % (w/w)
- . flavours 0 to 5 % (w/w)
- . eggs 0 to 20 % (w/w)
- . water 30 to 85 % (w/w)

Overrun:

Overrun is defined as in Ice Cream - W.S. Arbuckle - Avi Publishing - 1972 - page 194 .

5 Destabilising emulsifier

Destabilising emulsifier means any emulsifier which gives, at a level of 0.3%, a level of extracted fat of at least 25% in an ice cream premix containing 12% butter oil, 13% skim milk powder and 15% sucrose as described in on Figure 10 4 in 'The stability of aerated milk protein emulsions in the presence of small molecule surfactants' 1997 - Journal of Dairy science 80:2631:2638.

Examples of such destabilising emulsifiers are unsaturated 15 monoglyceride, polyglycerol esters, sorbitan esters, stearyl lactylate, lactic acid esters, citric acid esters, acetyllated monoglyceride, diacetyl tartaric acid esters, polyoxyethylene sorbitan esters, lecithin and egg yolk.

20 MethodsIce cream premix production

In a jacketed 500 litre mix tank, water is added at 85°C, then milk powder, sugar, stabilizers, butteroil with 25 emulsifier dissolved are added and mixed with high shear mixer and heated to maintain a temperature of 65°C for standard production and 55°C for production according to the invention:

30 Standard production: the premix is heated with plate heat exchanger to 83°C, homogenize with Crepaco single stage valve homogeniser at 140bar. After holding at 83°C for 15 seconds

- 6 -

the mix was cooled with a plate heat exchanger to 5°C and held at this temperature for at least two hours prior to freezing.

- 5 Invention: the premix was heated with a plate heat exchanger to 83°C and held at this temperature for 15 seconds to pasteurize the mix. The mix was tempered at 55°C (+/- 5°C) in a holding tank prior to homogenization and collected after a single pass through the homogeniser (Nanojet
- 10 Impinging Jet, ref: Verstallen, A., Apparatus for homogenizing essentially immiscible liquids for forming an emulsion described in Patent No. US5366287) at an input pressure of 1600bar (+/- 50bar). During homogenization there is a temperature rise of 2-2.5°C/100bar. Immediately
- 15 after homogenization the mix is passed through a plate heat exchanger and cooled to 8°C (+/- 3°C). The mix is held in a jacketed aging vessel at this temperature for at least two hours prior to freezing.

20 Ice cream Processing

The mix was processed according to two different routes.

Standard freezer

- The mix was aged overnight and was processed through an ice
- 25 cream freezer (Crepaco W104 freezer (SSHE) with a series 80 dasher operating at 4 bar barrel pressure). All ice cream was produced at a mix throughput of 120 l/hr at 60 % or 100% overrun with an extrusion temperature of -6.0°C and -5°C respectively. Ice cream was collected in 500 ml waxed paper
- 30 cartons and hardened in a blast freezer at -35°C for two hours.

Single Screw Extruder

- 7 -

The outlet of the SSHE was connected to a single screw extruder (SSE) (as described in WO98/09534) resulting in exit temperatures of ca. -14.5°C.

5 Tests

Fat composition

Fat composition analysis was carried out according to the Rose-Gottlieb method: British Standard Methods for Chemicals
10 analysis of ice cream, Part 3. Determination of fat content (BS2472: part 3: 1989 ISO 7328-1984).

Pieces of ice cream are randomly selected to give a total mass of approximately 100g, placed in a blender jar, covered
15 with a lid and allowed to soften at room temperature. This mix is then blended for two minutes (up to 7 minutes for products containing particulates, e.g. nuts) to obtain a homogeneous mixture. The temperature is kept below 12 C during softening and blending. 4 to 5 g (accurately measured
20 to 1mg) are weighed into a fat extraction flask and water at 65 C is added to obtain a total volume of 10ml and mixed thoroughly. Ammonia solution (2ml, 25% (m/m) of NH₃) is added and the flask immediately heated at 65 C for 15-20 minutes in a water bath and cooled to room temperature at
25 which time ethanol (10 ml) is added. Diethyl ether (25ml) is added and the flask shaken vigorously for 1 minute. Light petroleum (25ml) is the added and the flask shaken for 30 seconds. The stoppered flask is allowed to stand for 30
30 minutes before decanting the supernatant. The solvent is then removed by evaporation or distillation. The fat content is expressed as a percentage by weight.

Gas cell sizing

- 8 -

The microstructure of all ice cream samples was visualized by Low Temperature Scanning Electron Microscopy (LTSEM). All samples were stored at -80°C prior to structural analysis using a JSM 6310F scanning electron microscope fitted with an Oxford Instruments ITC4 controlled cold stage. The samples were prepared using the Hexland CP2000 preparation equipment. A sample at -80°C of size $5 \times 5 \times 10$ mm was taken from the centre of a 500 ml block of ice cream. This sample was mounted onto an aluminium stub using OCT mountant on the point of freezing and plunged into nitrogen slush. OCT is an aqueous based embedding medium used primarily for cryotome preparation of material for light microscopy. It is also called tissue tek and is supplied by Agar Scientific. The advantage of using oct rather than water to mount the samples for electron microscopy is that when OCT changes from liquid to solid ie. freezes it changes to opaque from transparent allowing visual identification of the freezing point. Identification of this point allows the sample to be mounted using a liquid at its coldest just prior to solidifying which will give support during rapid cooling. The sample was warmed to -98°C fractured and allowed to etch for 2 minutes before cooling to -115°C . The surface was coated with Au/Pd at -115°C , 6mA and 2×10^{-1} mBar Argon. The sample was transferred in vacuum to the LTSEM and examined under microscope conditions of -160°C and 1×10^{-8} Pa.

The gas structure in ice cream was quantified by measuring the gas cell size distribution from SEM images using the AnalySIS 2.11 - package AUTO (SIS Munster, Germany) with 'B' version software. The AnalySIS programme may be run using SEM images in two data formats, either as data direct from the JEOL microscope or as images scanned from Polaroids. All gas

- 9 -

cell sizes were measured from SEM micrographs. The optimum magnification was such that there were less than 300 gas cells per image. The programme was used semi-automatically such that particle edges were calculated automatically (by difference in grey-scale) and refined manually (by deleting and redrawing around particle boundaries not selected correctly). Since ice crystals may also have been selected by the programme, the gas cells were then manually selected and the distribution analyzed using the maximum diameter parameter. All gas cells present on an SEM micrograph were counted and up to six SEM images were used. Generally, at least 1000 gas cells were counted. The average size was determined as the number average, $d(1,0)$, of the individual cell sizes.

15

Premix fat droplet sizing

Particle sizes in the premix emulsion were measured using a Malvern Mastersizer (Malvern Instruments, UK) with water as the continuous phase using the 45mm lens and the presentation code 2 NAD. Ultrasound was applied to the Mastersizer tank for one minute before measurement. The surface weighted mean $d[3,2]$ was calculated. The diameter by which 90% by volume of the distribution was smaller, $d[0.9]$ was taken as the limit of individual fat droplets.

25

Ice cream fat droplet and fat aggregates

Two different methods were used.

Mastersizer Method:

20ml sample of ice cream was heated to 60°C for 5 minutes, added to the Malvern Mastersizer water bath, then sonicated for 2 minutes. The average droplet size, $d[3,2]$ and size distribution were measured. The proportion of fat aggregates

- 10 -

in the melted ice cream was calculated as the proportion of fat (expressed as % volume) with a particle size greater than the $d[0.9]$ determined for the unaggregated premix fat droplets.

5

Solvent Extraction Method:

10g sample (W1) is weighed into a measuring cylinder and left at room temperature to melt for 4 hours. 50ml petroleum spirit is added, the cylinder stoppered and inserted into a mechanical agitator. The cylinder is inverted for one minute at a rate of one inversion per second and then allowed to stand for 5 minutes and the solvent decanted in to a pre-weighed beaker (W2). A further 25ml solvent is added and the cylinders inverted 3 times by hand. After standing (2-3 minutes) the solvent layer is decanted again into the beaker. The beaker is placed in a fume cupboard overnight to evaporate the solvent and then dried in a spark proof oven at 100 C for 15-30 minutes. The beaker is then cooled in a dessicator and reweighed (W3). The percentage of de-emulsified fat is $[(W3-W2)/(C \times W1)] \times 100$ where C is the percentage of fat in the ice cream divided by 100.

General description of the Invention

25 It is a first object of the present invention to propose a process for manufacturing a frozen aerated product having an overrun of between 20% and 180%, preferably between 60% and 100%, comprising the steps of;

30 . producing a premix comprising 2 to 15 % fat (w/w), up to 1% (w/w) emulsifier, and 45 to 85 % (w/w) of water,

. homogenizing the premix in order to produce fat droplets having a $d(3,2)$ below 0.6 micron, preferably below 0.5 micron, even more preferably below 0.4 micron,

- 11 -

. cooling, freezing and aerating the homogenised premix. The product can then be extruded and optionally deep frozen.

This enables the production of smaller fat droplets which in turn generate smaller air cells, preferably wherein the mean gas cell size $d(1,0)$ is below 20 micron, more preferably below 10.5 micron. It has also been found that out of two ice creams with the same composition and the same air cell size, the one with the smallest fat droplets was found to be the preferred one when tasted by a trained panel.

In a first preferred embodiment of the invention the homogenising step takes place at a pressure of between 1000 and 2000 bar, preferably between 1400 and 1800 bar.

In a second preferred embodiment of the invention, the premix contains a destabilising emulsifier. Preferably the destabilising emulsifier is selected within the group consisting in unsaturated monoglyceride, polyglycerol esters, sorbitan esters, stearyl lactylate, lactic acid esters, citric acid esters, acetyllated monoglyceride, diacetyl tartaric acid esters, polyoxyethylene sorbitan esters, lecithin and egg yolk. More preferably the destabilising emulsifier is unsaturated monoglyceride. Preferably also the (destabilising emulsifier/fat) weight ratio of the premix is between 10:1500 and 15:300, even more preferably between 15:1200 and 15:600.

The incorporation of destabilising emulsifier, and particularly unsaturated monoglycerides, allows for the production in a SSHE of a frozen aerated product with gas cells smaller than the one obtained by freezing a premix in a

- 12 -

SSHE followed by cold extrusion in a SSE as disclosed in WO98/09534.

In a third preferred embodiment of the invention, the
5 homegenized premix is first frozen at a temperature of
between -4 C and -7 C in a scrapped surface heat exchanger
and then extruded in a screw extruder at a temperature of
between -10 C and -18 C. Even more preferably, the screw
extruder is a single screw extruder.

10

The combination of Ultra High Pressure homogenization
together with cold extrusion, allows the production of an
aerated product product with gas cells smaller than the one
obtained by freezing a premix in a SSHE followed by cold
15 extrusion in a SSE as disclosed in WO98/09534.

15

Preferably also, the temperature of the premix prior to
homogenisation is above 50 C. More preferably, the
homogenisation generates a temperature rise of the premix of
20 between 30 C and 45 C. By so doing it is no longer necessary
to use a plate-pack heat exchanger for pasteurisation.
Moreover, by starting with a temperature of the premix, prior
to homogenization of above 50 C while having a temperature
rise of below 45 C, it is possible to reach a temperature
25 after homogenisation which is not above 95 C, something which
prevents the water from boiling, something which would
generate bubbles in the premix.

25

Before, or after homogenization, it is possible to have a
30 pasteurization step.

30

It is a second object of the present invention to provide a
frozen aerated product, having an overrun of between 20% and

180%, preferably between 60% and 100%, and comprising 2 to 15% (w/w) of fat and destabilising emulsifier in a (destabilising emulsifier /fat) weight ratio of between 10:1500 and 15:300, preferably between 15:1200 and 15:600.

5

Preferably the destabilising emulsifier is selected within the group consisting in unsaturated monoglyceride, polyglycerol esters, sorbitan esters, stearyl lactylate, lactic acid esters, citric acid esters, acetyllated

10

monoglyceride, diacetyl tartaric acid esters, polyoxyethylene sorbitan esters, lecithin and egg yolk. More preferably the destabilising emulsifier is unsaturated monoglyceride.

15

More preferably the (destabilising emulsifier/fat) weight ratio of the frozen aerated product is between 10:1500 and 15:300, even more preferably between 15:1200 and 15:600.

20

It is a third object of the present invention to provide a frozen aerated product having an overrun of between 20% and 180%, preferably between 60% and 100%, and comprising 2 to 15% (w/w) of fat, wherein the mean gas cell size $d(1,0)$ is below 10.5 micron.

25

Detailed description of the Invention

The present invention will be further described in the following examples.

30

Example 1:

In this example various premixes were produced, homogenised and pasteurised according to the prior art and then processed

- 14 -

in a SSHE, some of the samples produced in the SSHE being then processed in a SSE.

The various premixes had the following composition (the composition are indicated in % w/w)

	A	B	C	D	E	F
Emulsifier	0.39	0.39	0.39	0.39	0.39	0.3
(*)						
Corn Syrup		2.2904	3.3861	4.4818	6.6732	6
Stabiliser	0.2	0.2	0.2	0.2	0.2	0.22
Whey protein	2.6	2.6	2.6	2.6	2.6	
Sucrose	15.6	15.1878	14.9906	14.7933	14.3989	13
Skimmed Milk Powder	7.4	7.4	7.4	7.4	7.4	13
butter fat	12.1	10	9	8	6	3
Flavour	0.254	0.254	0.254	0.254	0.254	0.12
Water	61.456	61.6778	61.7793	61.8809	62.0839	64.36

(*)Admul MG 4223 (referred as MGP in the rest of the description) which is a mono/diglyceride prepared from edible vegetable oil and commercially available from Quest International.

The above premixes were then homogenised and pasteurised at a temperature of 81-84 C for about 12 seconds at a pressure of 140 bar.

The obtained pre-mixes were then processed in a SSHE under the following conditions. Ice creams at a temperature of -6 C (+/- 0.1 C) with an overrun of 60% (+/- 1%) were produced.

- 15 -

	A	B	C	D	E	F
Input temperature	8.8 C	12.8 C	16.2 C	8 C	14.5 C	9.5C
Output Temperature	-6.1 C	-6 C	-6 C	-6.1 C	- 6.1 C	-6.2 C
overrun	61%	60%	60%	61%	61%	60%
barrel pressure	4 bar	4.06 bar	3.99 bar	4 bar	3.98 bar	4 bar

Part of the samples A,B,C,D,E and F were then processed in a single screw extruder at an input temperature of -6C giving an extruded product at a temperature of between -14C and -15C.

Example 2:

10

The premixes A, B, C, D,E and F of Example 1 were pasteurised at a temperature of 81-86 C for 12 seconds.

The pasteurised premixes were then homogenised in a Nanojet 200/2000 (commercially obtainable from Nanojet - Germany). A detailed description of such a homogeniser can be found in US5,366,287. The premixes were input into the homogeniser at a temperature of 54-58 C and treated at a pressure of 1600 bar. The temperature at the outlet of the homogeniser was between 91 and 95 C.

The obtained homogenised premixes were then processed in a SSHE under the following conditions.

- 16 -

	A	B	C	D	E	F
Input temp	18.7 C	13.2 C	13.5 C	11.2 C	11.0 C	9.5 C
Output temp	-6.3 C	-5.8 C	-6.7 C	-6.0 C	- 6.4 C	-6.2C
overrun	60%	60%	61%	60%	60%	60%
barrel pressure	4 bar	4 bar	4.01 bar	4 bar	4 bar	4bar

Part of the samples A to F were then processed in a single screw extruder and produced ice cream at a temperature of 5 between -14 C and -15 C.

Example 3:

The premix compositions in example 1 were modified, the emulsifier being now a blend of Admul MG 4223 (0.24 % w/w based on the total weight of the premix) and H7804 (0.15 % w/w based on the total weight of the premix) for samples F,G,I and J and a blend of Admul MG 4223 (0.265 % w/w based on the total weight of the premix) and H7804 (0.125 % w/w based on the total weight of the premix) for sample H. H7804 is an unsaturated monoglyceride commercially available from Quest International. Admul MG 4223 (referred as MGP in the rest of the description) is a mono/diglyceride prepared from edible vegetable oil and commercially available from Quest International.

	F	G	H	I	J
Emulsifier (MGP+H7804)	0.39	0.39	0.39	0.39	0.39
Corn Syrup		2.2904	3.3861	4.4818	6.6732
Stabiliser	0.2	0.2	0.2	0.2	0.2
Whey protein	2.6	2.6	2.6	2.6	2.6
Sucrose	15.6	15.1878	14.9906	14.7933	14.3989
Skimmed Milk Powder	7.4	7.4	7.4	7.4	7.4
butter fat	12.1	10	9	8	6
Flavour	0.254	0.254	0.254	0.254	0.254
Water	61.456	61.6778	61.7793	61.8809	62.0839

The obtained premixes were then pasteurised at a temperature of 81-85 C for 12 seconds and homogenised as in example 2.

- 5 The premixes were input into the homogeniser at a temperature of 55-65 C and treated at a pressure of 1600 bar. The temperature, at the outlet of the homogeniser was between 89 and 92 C.

- 10 The pasteurized and homogenized premixes were then processed in SSHE under the following conditions.

	F	G	H	I	J
Input temperature	13 C	16 C	16.9 C	11.5 C	11.7 C
Output Temperature	-5.7 C	-5.9 C	-6.0 C	-6.0 C	- 6.1 C
Overrun	60%	60%	60%	59.5%	59 %
barrel pressure	4.01 bar	4 bar	4 bar	4 bar	3.98 bar

Fat droplets particle size

- 15 Fat droplets particle of each premix after homogenisation was measured. The results are summarized in the following table.

Fat Content (%)	3	6	8	9	10	12
Example 1	0.42	0.45	0.51	0.53	0.50	0.53
d(3,2) (microns)						
Example 1	1.24	1.19	1.36	1.44	1.56	1.59
d(0,9) (microns)						
Example 2	0.34	0.34	0.39	0.39	0.36	0.35
d(3,2) (microns)						
Example 2	0.9	0.83	1.14	1.64	0.94	1.03
d(0,9) (microns)						
Example 3		0.34	0.32	0.31	0.36	0.35
d(3,2) (microns)						
Example 3		0.83	0.94	0.72	0.86	0.92
d(0,9) (microns)						

Aggregated fat

The percentage of aggregated fat was measured both using the Mastersizer method and the solvent extraction method.

5

Mastersizer method

Fat Content (%)	3	6	8	9	10	12
Example 1 SSHE	8.24	20.69	28.60	37.05	17.44	20.02
Example 1 SSE		26.97	41.78	53.80	64.47	66.98
Example 2	18.8	25.46	37.88	39.46	54.12	62.56
Example 3		52.13	66.20	62.44	68.45	80.10

Solvent Extraction

Fat Content (%)	3	6	8	9	10	12
Example 1 SSHE	7.6	10.04	21.38	22.2	23.27	15.44
Example 1 SSE	3.96		37.83	50.54	66.66	65.48
Example 2	3.18	4.20	3.01	6.10	3.17	6.69
Example 3		8.19	26.54	18.12	5.57	46.88

10 By comparing the results generated by the two methods, it can be seen that, owing to the small size of the droplets in examples 2 and 3, the results which are obtained when using solvent extraction are not consistent, and show no trend. It

is due to the fact that it is difficult for the solvent to extract the fat from agglomerates made out of small fat droplets. It is the reason why, in order to characterize the products of the present invention, the first method
 5 (Mastersizer) is preferred.

Mean gas cell size:

The mean gas cell size (d(1,0)) of all the samples was measured in microns, the results are summarized herebelow.

10

Fat Content (%)	3	6	8	9	10	12
Example 1 SSHE	66.57	31.35	20.49	24.39	23.38	18.8
Example 1 SSE	12.00	12.28	10.31	12.51	11.49	12.8
Example 2	10.3	9.42	8.42	9.71	10.4	9.81
Example 3	9.49	9.4	9.59	9.46	9.86	8.99

15

Example 1 shows that, except at high fat content (above 10%), it is not possible using a standard SSHE to produce a product with a mean gas cell size of below 20 microns when using MGP as emulsifier system whereas this is a standard emulsifier used in the ice cream industry. It is only by using a specific type destabilising emulsifier that when using a standard SSHE a mean gas cell size of below 20 microns is achieved.

Claims

1. Process for manufacturing a frozen aerated product having an overrun of between 20% and 180%, preferably between 60% and 100%, comprising the steps of;
5
 . producing a premix comprising 2 to 15 % fat (w/w), up to 1% (w/w) emulsifier, and 45 to 85 % (w/w) of water,
 . homogenizing the premix in order to produce fat droplets having a $d(3,2)$ below 0.6 micron, preferably below 0.5
10 micron, even more preferably below 0.4 micron,
 . cooling, freezing and aerating the homegenised premix.
2. Process according to claim 1 wherein the homogenising step takes place at a pressure of between 1000 and 2000 bar,
15 preferably between 1400 and 1800 bar.
3. Process according to claim 1 wherein the premix contains a destabilising emulsifier.
- 20 4. Process according to claim 3 wherein the destabilising emulsifier is selected within the group consisting in unsaturated monoglyceride, polyglycerol esters, sorbitan esters, stearyl lactylate, lactic acid esters, citric acid esters, acetyllated monoglyceride, diacetyl tartaric
25 acid esters, polyoxyethylene sorbitan esters, lecithin and egg yolk.
5. Process according to claim 4 wherein the (destabilising emulsifier/fat) weight ratio of the premix is between
30 10:1500 and 15:300, even more preferably between 15:1200 and 15:600.
6. Process according to claim 1 wherein the homegenized premix is first frozen at a temperature of between -4 C

- 21 -

and -7 C in a scrapped surface heat exchanger and then extruded in a screw extruder at a temperature of between -10 C and -18 C.

- 5 7. Process according to any preceding claim wherein the temperature of the premix prior to homogenisation is above 50 C.
8. Process according to claim 7 wherein the homogenising step
10 generates a temperature rise of the premix of between 30 C and 45 C. Before, or after homogenization, it is possible to have a pasteurization step.
9. Frozen aerated product, having an overrun of between 20%
15 and 180 %, preferably between 60% and 100%, and comprising 2 to 15 % (w/w) of fat and destabilising emulsifier in a (destabilising emulsifier /fat) weight ratio of between 10:1500 and 15:300, preferably between 15:1200 and 15:600.
- 20 10. Frozen aerated product according to claim 9 wherein the destabilising emulsifier is selected within the group consisting in unsaturated monoglyceride, polyglycerol esters, sorbitan esters, stearyl lactylate, lactic acid esters, citric acid esters, acetyllated monoglyceride,
25 diacetyl tartaric acid esters, polyoxyethylene sorbitan esters, lecithin and egg yolk.
11. Frozen aerated product according to claim 10 wherein
30 wherein the (destabilising emulsifier/fat) weight ratio of the frozen aerated product is between 10:1500 and 15:300, even more preferably between 15:1200 and 15:600

- 22 -

12. Frozen aerated product having an overrun of between 20% and 180 %, preferably between 60% and 100%, and comprising 2 to 15 % (w/w) of fat, wherein the mean gas cell size $d(1,0)$ is below 10.5 micron.

5

13. Frozen aerated which presents more than 5% of its fat (by weight on the total fat weight) in aggregate form (as measured by the Mastersizer method), preferably more than 40%, more preferably more than 50%.

INTERNATIONAL SEARCH REPORT

International Application No

PC, EP 99/04737

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23G9/00 A23G9/04 A23G9/20

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	<p>OLSON D W ET AL: "Role of microfluidization in the manufacture of frozen dairy desserts." MISSISSIPPI STATE MS39762, USA, vol. 78, no. Suppl. 1, 1995, page 149 XP002090714 Dep. of Food Sci. & Tech., Southeast Dairy Foods Res. Cent., Mississippi Agric. & Forestry Exp. Sta., Mississippi State Univ., Mississippi State, MS 39762, USA abstract</p>	1-5,7-12
Y	<p>EP 0 455 288 A (UNILEVER NV ;UNILEVER UK CENTRAL RESOURCES (GB)) 6 November 1991 (1991-11-06) page 2, line 38 - line 43 page 2, line 58 -page 3, line 2; example 2</p>	1-5,7-12



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

5 November 1999

Date of mailing of the international search report

15/11/1999

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
 NL - 2280 HV Rijswijk
 Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
 Fax: (+31-70) 340-3016

Authorized officer

Guyon, R

INTERNATIONAL SEARCH REPORT

International Application No

PC 17 EP 99/04737

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 928 648 A (STAHL HOWARD D ET AL) 23 December 1975 (1975-12-23) the whole document ----	13
X	US 3 924 018 A (SIMS REX J ET AL) 2 December 1975 (1975-12-02) the whole document ----	13
A	WO 98 09536 A (UNILEVER) 12 March 1998 (1998-03-12) the whole document ----	1,3-6
A	US 4 725 445 A (FERRERO) 16 February 1988 (1988-02-16) the whole document ----	1,3-6
A	EP 0 469 656 A (UNILEVER NV ;UNILEVER PLC (GB)) 5 February 1992 (1992-02-05) the whole document ----	1-5,7-12
A	EP 0 593 833 A (NIRO SOAVI S P A) 27 April 1994 (1994-04-27) the whole document ----	1,2
X	WO 98 18350 A (UNILEVER PLC ;UNILEVER NV (NL)) 7 May 1998 (1998-05-07) claims; examples ----	1,2,6
A	US 5 486 372 A (MARTIN ROBERT W ET AL) 23 January 1996 (1996-01-23) examples 1,3 ----	1,2
A	DE 42 26 255 A (APV GAULIN GMBH) 10 February 1994 (1994-02-10) column 1, line 25 - line 54 column 2, line 26 - line 53; claims ----	1,2,13
A	EP 0 713 650 A (NESTLE SA) 29 May 1996 (1996-05-29) ----	
A	WO 92 09209 A (UNILEVER PLC ;UNILEVER NV (NL)) 11 June 1992 (1992-06-11) claims 1,8; examples 4.1-4.7 ----	1
A	US 5 472 726 A (GOOD HUMOR CORP) 5 December 1995 (1995-12-05) the whole document ----	1
A	EP 0 147 483 A (PILLSBURY CO) 10 July 1985 (1985-07-10) the whole document ----	13
A	US 4 434 186 A (DESIA NITIN ET AL) 28 February 1984 (1984-02-28) the whole document ----	13
	-/--	

INTERNATIONAL SEARCH REPORT

International Application No

PCT, EP 99/04737

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 956 519 A (EVANS MERVYN THOMAS ARTHUR ET AL) 11 May 1976 (1976-05-11) -----	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT, EP 99/04737

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0455288	A	06-11-1991	AT 96283 T	15-11-1993
			AU 627994 B	03-09-1992
			AU 7594191 A	07-11-1991
			CA 2041569 A,C	03-11-1991
			DE 69100553 D	02-12-1993
			DE 69100553 T	24-02-1994
			DK 455288 T	21-02-1994
			ES 2046005 T	16-01-1994
			FI 912099 A	03-11-1991
			JP 7067567 A	14-03-1995
			PT 97546 A	28-02-1992
			US 5149557 A	22-09-1992
			US 3928648	A
GB 1475425 A	01-06-1977			
US 3924018	A	02-12-1975	CA 1038222 A	12-09-1978
			DE 2526523 A	02-01-1976
			FR 2274237 A	09-01-1976
			GB 1491310 A	09-11-1977
WO 9809536	A	12-03-1998	AU 4206797 A	26-03-1998
			AU 4380997 A	26-03-1998
			WO 9809534 A	12-03-1998
US 4725445	A	16-02-1988	IT 1179107 B	16-09-1987
			AT 38610 T	15-12-1988
			AU 576391 B	25-08-1988
			AU 4864985 A	08-04-1986
			CA 1249165 A	24-01-1989
			DE 3531330 A	24-04-1986
			DE 3566212 A	22-12-1988
			WO 8601688 A	27-03-1986
			EP 0192753 A	03-09-1986
			HK 77793 A	13-08-1993
			SG 97191 G	19-02-1993
EP 0469656	A	05-02-1992	AT 116108 T	15-01-1995
			AU 667027 B	07-03-1996
			AU 8136691 A	06-02-1992
			CA 2048092 A,C	31-01-1992
			DE 69106261 D	09-02-1995
			DE 69106261 T	24-05-1995
			ES 2067846 T	01-04-1995
			FI 913624 A	31-01-1992
			JP 2025979 C	26-02-1996
			JP 4229150 A	18-08-1992
			JP 7048987 B	31-05-1995
			PT 98482 A,B	29-05-1992
			US 5336514 A	09-08-1994
EP 0593833	A	27-04-1994	NONE	
WO 9818350	A	07-05-1998	AU 6907498 A	22-05-1998
			EP 0936878 A	25-08-1999
US 5486372	A	23-01-1996	AU 1840995 A	25-09-1995
			CA 2183168 A	14-09-1995

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT, EP 99/04737

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5486372 A		WO 9524132 A	14-09-1995
DE 4226255 A	10-02-1994	NONE	
EP 0713650 A	29-05-1996	AT 177909 T	15-04-1999
		AU 1471999 A	15-04-1999
		AU 703449 B	25-03-1999
		AU 3770095 A	30-05-1996
		BR 9505269 A	21-10-1997
		CA 2163284 A	24-05-1996
		CN 1132595 A	09-10-1996
		CZ 9503076 A	17-07-1996
		DE 69508529 D	29-04-1999
		DE 69508529 T	09-09-1999
		ES 2130520 T	01-07-1999
		FI 955594 A	24-05-1996
		HU 74491 A	28-01-1997
		JP 8205783 A	13-08-1996
		NO 954687 A	24-05-1996
		NZ 280507 A	26-01-1998
		PL 311434 A	27-05-1996
		SG 35034 A	01-02-1997
		SK 144495 A	05-06-1996
		TR 960494 A	21-07-1996
		US 5919510 A	06-07-1999
WO 9209209 A	11-06-1992	AT 108306 T	15-07-1994
		AU 663061 B	28-09-1995
		AU 8863291 A	25-06-1992
		CA 2082543 A	13-05-1993
		CA 2096429 A,C	24-05-1992
		DE 69102885 D	18-08-1994
		DE 69102885 T	10-11-1994
		DK 558523 T	28-11-1994
		EP 0558523 A	08-09-1993
		ES 2056665 T	01-10-1994
		FI 932290 A	24-06-1993
		GR 3025710 T	31-03-1998
		IE 65342 B	18-10-1995
		JP 2620989 B	18-06-1997
		JP 6502530 T	24-03-1994
		KR 126819 B	26-12-1997
		MX 9102187 A	08-07-1992
		NZ 240627 A	22-12-1994
		PT 99588 A,B	30-10-1992
		TR 26436 A	15-03-1995
		US 5652011 A	29-07-1997
		ZA 9109231 A	21-05-1993
US 5472726 A	05-12-1995	AT 152583 T	15-05-1997
		AU 671703 B	05-09-1996
		AU 5694794 A	22-06-1994
		BR 9307573 A	15-06-1999
		CA 2150687 A	09-06-1994
		CN 1095555 A,B	30-11-1994
		CZ 9501431 A	13-12-1995
		DE 69310545 D	12-06-1997
		DE 69310545 T	11-09-1997

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT, EP 99/04737

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5472726 A		DK 675685 T	27-10-1997
		WO 9412050 A	09-06-1994
		EP 0675685 A	11-10-1995
		ES 2102189 T	16-07-1997
		FI 952694 A	02-06-1995
		GR 3023967 T	31-10-1997
		JP 8503608 T	23-04-1996
		NO 952179 A	28-07-1995
		NZ 258873 A	25-06-1996
		PL 309279 A	02-10-1995
		SK 73395 A	08-11-1995
	ZA 9309041 A	02-06-1995	

EP 0147483 A	10-07-1985	NONE	

US 4434186 A	28-02-1984	NONE	

US 3956519 A	11-05-1976	AU 7642074 A	17-06-1976
		DE 2458145 A	26-06-1975
		ES 432861 A	01-04-1977
		FR 2254280 A	11-07-1975
		JP 50116653 A	12-09-1975
		NL 7416095 A	17-06-1975
		BE 823285 A	12-06-1975
		ZA 7407897 A	25-08-1976
