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DIVISION DE NUEVAS CREACIONES
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
E. S. D.

COD:01

REF: EXPEDIENTE No.04084717
FECHA DE LA SOLICITUD: Agosto 30, 2004
SOLICITANTE: NATIONAL STARCH AND CHEMICAL
INVESTMENT HOLDING CORPORATION
TITULO: ALMIDÓN PARA POSTRES CONGELADOS

ASUNTO: PRESENTACIÓN DEL DOCUMENTO DE PRIORIDAD

DILIA RODRIGUEZ D'ALEMAN, en mi condición de apoderada de la sociedad solicitante, en esta oportunidad por el presente escrito me permito presentar:

- Copia certificada de la prioridad Estadounidense reivindicada N° 60/511.765 de Octubre 16 de 2003.
- Su traducción al español.
- Copia certificada de la prioridad Estadounidense reivindicada N° 60/498.834 de Agosto 29 de 2003.
- Su traducción al español.
- Copia certificada de la prioridad Estadounidense reivindicada N° 10/926.555 de Agosto 26 de 2003.
- Su traducción al español.

Esta prioridad está siendo presentada dentro del término de 16 meses que exige la Decisión 486 de Diciembre 1 de 2.000.

Ruego a usted continuar con el presente trámite.

Señor Jefe,


DILIA RODRIGUEZ D'ALEMAN
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6	DISQUETES		
7	SOBRE DE MANILA		
8	SOBRE BLANCO TAMAÑO CARTA		
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10	OTROS:		

Observaciones: Arte final

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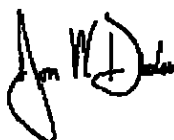
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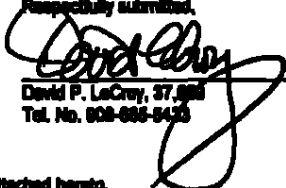
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PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53 (b)(2).

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TITLE OF THE INVENTION (250 characters max)					
STARCH FOR FROZEN DESSERTS					
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ENCLOSED APPLICATION PARTS (check all that apply)					
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☒ Drawing(s)		Number of Sheets: 3		☐ Other (specify) Postcard Receipt	
METHOD OF PAYMENT					
The Commissioner is hereby authorized to charge filing fees and additional fees required to Deposit Account Number: 14-8488.				PROVISIONAL FILING FEE AMOUNT (\$): 180.00	

- ☒ The invention was not made by an agency of the United States Government or under a contract with an agency of the United States Government
- ☐ A Power of Attorney accompanies this cover sheet.

Respectfully submitted,

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Date: October 18, 2003

- ☐ Additional inventors are being named on separately numbered sheets attached hereto.

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STARCH FOR FROZEN DESSERTS

BACKGROUND OF THE INVENTION

Technical Field. The present invention relates to a starch for use in frozen food products. More specifically, the present invention is directed towards a starch or starch derivative for use in frozen desserts in order to obtain improved structure characteristics, including slow melt down, excellent shape retention, reduced iciness and greater heat shock stability.

Background Information. In addition to their rich flavor, frozen confectioneries are enjoyed for their creaminess and smoothness. However, in order to preserve these characteristics, these products have to be handled and stored with care. Unfortunately, even small temperature variations can occur during storage, distribution and handling. For example, such variations can occur when a consumer buys a frozen product and does not consume it right away, as in the time from when a consumer purchases such a product at a grocer and places that product in his freezer. Partial or even substantial defrosting of the product can occur before it is refrozen. This temperature cycling can result in ice crystal growth in the product, as well as loss in product shape. Such growth affects both the visual appearance and organoleptic properties of the frozen product, thereby reducing its quality and appeal, at least as perceived by the consumer. The most frequently occurring textural defect in ice cream is due to ice crystal growth, and is the primary limitation to its shelf life. As such, it is desirable for frozen desserts to be stable against 'heat shock', or the cyclic conditions of partial thawing and refreezing that occur during typical storage, shipping and handling of these products.

As used herein, a frozen dessert, frozen confectionery or frozen product refers to a product in which water is present in both the liquid and frozen state. Examples of frozen desserts include ice cream, ice milk, water ice and parfaits. As used herein, frozen novelties refer to frozen desserts such as those available on a stick or in sandwiches or cones.

Ice crystals in frozen products need to be numerous and of small, nearly uniform size to avoid detection when eaten. Because ice crystals are relatively unstable, during storage they undergo changes in number, size and shape, which is collectively referred to as recrystallization. While some recrystallization occurs naturally at constant temperatures, by far the majority of it occurs due to temperature fluctuations or heat shock. An increase in temperature during the frozen storage of a product such as ice cream causes some of the ice crystals, particularly the

smaller ones, to melt. As the temperature decreases, that water refreezes without renucleation, instead depositing on the surface of larger crystals. As such, heat shock or temperature cycling in frozen products reduces the total number of ice crystals and increases the mean ice crystal size.

To a consumer, the perception of coldness and iciness of ice cream is directly related to the amount of ice formed in the ice cream and the size of the ice crystals. Typically, consumers can detect ice crystals larger than about fifty (50) microns. Such large ice crystals contribute to the coarseness and iciness of the ice cream. For a typical ice cream, the amount of ice formed and the crystal size distribution are mainly determined by the formulation, processing, and storage conditions as outlined below.

Ice crystallization is also affected by the glass transition temperature (T_g) and the freezing point of the ice cream. In a maximally freeze-concentrated solution, an invariant condition occurs at temperature T_g and concentration C_g (or unfreezable water W_g), depending on the particular mixture of compounds in the formulation. The unfreezable water W_g is that water rendered unfreezable in the maximum freeze-concentrated solution due to the low diffusivity in the glassy state. When ice cream is stored below T_g , the system is in the glassy state. In this glassy state, the diffusion-controlled processes that typically result in reduced quality and stability can be largely inhibited. Above T_g , the system is in a rubbery state where the translational diffusion is free to occur. The rate of diffusion-controlled deterioration increases exponentially with increasing ΔT , the temperature difference between storage temperature T and T_g ($\Delta T = T - T_g$), in agreement with William-Landel-Ferry ('WLF') kinetics. Practically speaking, the higher the W_g , the less water there is available for freezing at the same solid content. For frozen desserts such as ice cream, a higher W_g means less iciness.

Various stabilizing gums have been used as additives in an attempt to improve the heat shock stability and creaminess of frozen food products. Such stabilizing gums have included gelatin, agar, gum acacia, guar gum, carob or guar seed flour, locust bean gum, carrageenan, alginate, carboxymethyl cellulose, xanthan and the like, with each exhibiting their own set of advantages and disadvantages. Microcrystalline cellulose ("MCC") and carboxymethyl cellulose are often used in combination with other stabilizing gums to improve functional effectiveness. MCC and cellulose gum can be used together to reduce ice crystal growth. However, MCC can

continue to activate during aging or subsequent processing, resulting in unanticipated viscosity. MCC can also be costly and prevent frozen products from carrying a 'natural' label.

While gums improve stability, their use has several disadvantages. For example, the amount of stabilizing gum required to provide heat shock stability can result in a product having an unacceptably greasy and/or gummy mouthfeel. Further, products containing such stabilizing compounds are regulated and often poorly perceived by the public. In addition, these additives can be expensive. Several attempts have been made to improve heat shock stability of frozen products by replacing at least part of the stabilizer(s) in the product with other components. However, stabilizers have the added benefit of reducing the size of ice crystals in those products. By replacing all or part of those stabilizers, the size of ice crystals in the product can increase, resulting in a less desirable product.

Emulsifiers provide stability and creaminess to frozen products by facilitating an interface between the aqueous phase and the fat phase of the product. Milk or milk proteins and eggs yolks are natural sources of emulsifiers useful for their water-binding properties. Commercially available emulsifiers are generally derived by chemical reactions with naturally occurring glycerides.

Emulsifiers added to ice cream reduce the stability of this fat emulsion by replacing proteins on the surface of the milkfat. For example, ice cream is a frozen dessert that is both an emulsion and foam. When an ice cream mixture is whipped, the fat emulsion begins to break down and the fat globules begin to flocculate or destabilize. This partially coalesced fat stabilizes air bubbles that are whipped into this mix. Without emulsifiers, the fat globules would be able to resist this coalescing due to the proteins being absorbed to the fat globule, causing the air bubbles to not be properly stabilized and affecting the texture or smoothness of the ice cream.

Emulsifiers also affect the melt down of the ice cream. When ice cream is placed in an ambient environment, the ice cream melts and the fat-stabilized foam structure collapses. Melting is controlled by outside temperature and rate of heat transfer. However, even after the ice crystals melt, the ice cream does not collapse until the fat-stabilized foam structure collapses. This collapse is a function of the extent of fat destabilization/partial coalescence, which is controlled by the emulsifier concentration.

The frozen product ice cream is basically composed of milkfat, serum solids or milk solids-not-fat, sweeteners, stabilizers and emulsifiers, and water. Stabilizers and emulsifiers

were previously discussed. Water found in ice cream comes from the milk or other ingredients or is added. The milkfat or butterfat is typically obtained from sweet or heavy cream, and provides flavor, smooth texture and body to the ice cream. Other types of fats, such as palm oil, can also be used. The serum solids contain the lactose, caseins, whey proteins, minerals and ash content, and can be found in concentrated skimmed milk or skim milk powder. Sucrose is typically the main sweetener. At least a portion of the sweetener can be obtained from corn syrup solids, which provides a firmer and chewier body to the ice cream. Liquid and solid corn syrup is available in various dextrose equivalents ("DE"), which is a measure of the reducing sugar content of the syrup calculated as dextrose and expressed as a percentage of the total dry weight. As the DE increases, the sweetness increases and the molecular weight decreases.

The formulation used for manufacturing frozen desserts varies depending upon the type of product desired. Types of frozen products include economy brands typically having the legal minimum fat and total solids content, standard brands, premium brands and super premium brands, as well as a hard frozen, soft frozen, low fat, light, sherbet, sorbet or frozen yogurt product. As the amount of fat increases in the formulation, the frozen product becomes less viscous.

Ice cream ingredients are blended together according to the formulation selected to produce the ice cream mix. The mix is then pasteurized, followed by homogenization, or vice versa. Once homogenized and pasteurized, the mix is aged to allow the fat to cool and crystallize, and the proteins and polysaccharides to fully hydrate. After aging, the mix is frozen, during which particulates such as fruit, nuts, candy, cookies, etc. may be added to the mix, followed by hardening of the ice cream, typically at temperatures of -30 to -40°C . The freezing and hardening is best done at a fast rate, as a fast freezing rate promotes the formation of many small ice crystals instead of fewer larger ice crystals.

The temperature at which ice cream freezes is affected or depressed by the concentration of sweeteners and/or their molecular weight. For example, a mix having a higher concentration of sugars and/or sugars of a lower molecular weight will have a lower freezing temperature curve than a mix having a lower concentration of sugars and/or sugars of a higher molecular weight. The lower the freezing curve, the softer the ice cream, the greater the freeze out of the water during hardening, and the greater the susceptibility of the ice cream to heat shock.

For low fat or reduced fat frozen desserts, particularly frozen novelties, shape retention is necessary for product quality. It is known that melt characteristics of frozen desserts are relevant to shape retention. For ice cream, melt down is strongly influenced by the fat and air domains of the ice cream. As explained above, both fat and emulsifiers in the dessert affect melt down. For a low or reduced fat formula, melt down can be critical. Simply adding more emulsifier to make up for the loss of fat can be expensive and affect the characteristics of the ice cream, and is therefore not a preferred solution.

Various starch-based products have been marketed and sold for use in frozen desserts. These include converted starches, stabilized starches and crosslinked starches. It is well known in the food industry to use starch as a thickener and/or binder, *i.e.*, as a stabilizer. Typically, starches have been used in frozen confectioneries as a fat substitute or texturing agent. With the trend toward diminishing sugar in frozen products such as ice cream, starch-based products such as hydrogenated starch hydrolysates (polyols) have proven useful alternatives to sweeteners such as maltodextrin and polydextrose. These starch sweetener substitutes are typically the liquid and/or solid corn syrups previously discussed. However, these substitutes do not function in reducing melt down and/or reducing iciness in frozen desserts.

Accordingly, there is a need for a starch product for use in frozen desserts that reduces the melt down of the dessert when exposed to temperature fluctuations during storage and distribution, thereby aiding in retaining the shape of the frozen product, particularly low fat products. Finally, there is a need for a starch product for use in frozen desserts that reduces the risk of ice crystal growth during temperature cycling.

SUMMARY OF THE INVENTION

The present invention is directed towards starch and/or starch derivatives useful in frozen desserts for obtaining improved structure characteristics, including a reduction in melt down, improved shape retention and/or decrease in ice crystal growth during temperature cycling. The invention is also directed towards a frozen dessert that includes those starch and/or starch derivatives. The frozen dessert can include fat, sweeteners, milk solids-not-fat, stabilizers, emulsifiers, water and starch. The starch and/or starch derivative of the present invention can be used to replace at least a portion of any of the other solids, or can be added on top of those solids.

BRIEF DESCRIPTION OF THE DRAWINGS

The manner in which these objectives and other desirable characteristics can be obtained is explained in the following description and attached drawings in which:

Figure 1 is a graph illustrating the percent melt down of various ice creams after four hours at 19°C.

Figure 2 is a graph illustrating the correlation between the melt down of ice cream and the $\tan \delta$ of the mix after eight days at -6.7°C.

Figure 3 is a photograph illustrating the effect in melt down of an ice cream containing a starch of the present invention after heat shock treatment as compared to freshly prepared ice cream.

Figure 4 is a melt down curve of an ice cream containing 3% of a starch of the present invention as compared to a control ice cream containing no starch.

DETAILED DESCRIPTION OF THE INVENTION

The starch base material used for the present invention may be derived from any source, including cereal or root starches. Typical sources for the starches are cereals, tubers, roots, legumes and fruits. Native starch sources include, for example, any variety of corn (maize), pea, potato, sweet potato, banana, barley, wheat, rice, oat, sago, amaranth, tapioca, arrowroot, canna, sorghum and waxy and high amylose varieties thereof. As used herein, "waxy" includes starches containing no more than about 10% amylose by weight. As used herein, the term "high amylose" includes starches containing at least about 40% by weight amylose. As used herein, the term "amylose-containing" includes those starches containing at least about 10% by weight amylose. Preferably, the starch base material is an amylopectin starch such as a waxy corn starch or amylose-containing starch.

The granular starch base can be one that has been lightly converted or hydrolyzed to a water fluidity ("WF") of about 0 to about 80. The term "water fluidity" has a very specific meaning as described further herein below. The starch is converted to its fluidity or thin-boiling form using a suitable method of degradation that results in the modified starch defined herein. Such degradation includes, for example, mild acid hydrolysis with an acid such as sulfuric or hydrochloric acid, conversion with hydrogen peroxide, or enzyme conversion. Converted starch products can include blends of different starches converted by various techniques as well as

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converted starch(es) blended with unconverted starch(es). Commercially, starch is typically converted by acid or enzyme conversion techniques. Hydrogen peroxide can also be used to convert or thin the starch, either alone or with metal catalysts.

As noted above, the starch of the present invention can be converted to a water fluidity ("WF") of from about 0 to about 80, particularly from about 0 to about 60. Water fluidity, as used herein, is an empirical test of viscosity measured on a scale of 0-90 wherein fluidity is inversely proportional to viscosity. Water fluidity of starches is typically measured using a rotational shear-type viscometer (commercially available from Thomas Scientific, Swedesboro, New Jersey), standardized at 30°C with standard oil that has a viscosity of 24.73 cps and requires 23.12 ± 0.05 sec for 100 revolutions. Accurate and reproducible measurements of water fluidity are obtained by determining the time which elapses for 100 revolutions at different solids levels depending on the starch's degree of conversion -- as the degree of conversion increases, the viscosity decreases and the WF values increase.

The base material can be modified either chemically or physically using techniques known in the art. The modification can be to the base or the converted starch, though typically the modification is carried out after conversion.

Physically modified starches, such as thermally inhibited starches described in International Publication WO 95/04082, may also be suitable for use herein. Physically modified starches are also intended to include fractionated starches in which there is a higher proportion of amylose.

Chemically modified starches include, without limitation, crosslinked starches, acetylated and organically esterified starches, hydroxyethylated and hydroxypropylated starches, phosphorylated and inorganically esterified starches, cationic, anionic, nonionic, and zwitterionic starches, carboxymethyl starch, and succinate and substituted succinate derivatives of starch. Such modifications are known in the art, for example in MODIFIED STARCHES: PROPERTIES AND USES, Ed. Wurzburg, CRC Press, Inc., Florida (1986).

When crosslinked, the starch is reacted with any crosslinking agent capable of forming linkages between the starch molecules. Typically crosslinking agents suitable herein are those approved for use in foods, such as epichlorohydrin, linear dicarboxylic acid anhydrides, acrolein, phosphorus oxychloride, and soluble metaphosphates. Preferred crosslinking agents are

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phosphorus oxychloride, epichlorohydrin, sodium trimetaphosphate (STMP), and adipic-acetic anhydride, and most preferably phosphorus oxychloride.

The crosslinking reaction itself is carried out according to standard procedures described in the literature for preparing crosslinked, granular starches. Examples of such art include U.S. Patent Nos. 2,328,537 and 2,801,242. Of course, the exact reaction conditions employed will vary with the type of crosslinking agent used, as well as the type of starch base, the reaction scale, etc. The reaction between the starch and the crosslinking agent can be carried out in aqueous medium. In this preferred method, the starch is slurried in water and adjusted to the proper pH, followed by addition of the crosslinking agent.

The amount of crosslinking agent necessary to give a product having the characteristics defined herein will vary depending on, for example, the water fluidity level of the starch, the type of pregelatinization employed, the type of crosslinking agent used, the concentration of the crosslinking agent, the reaction conditions, and the necessity for having a crosslinked starch that falls within a specified range of crosslinking as determined by its viscosity characteristics. One skilled in the art will recognize that it is not the amount of crosslinking agent added to the reaction vessel that determines the properties of the final product, but rather the amount of reagent that actually reacts with the starch, as measure by the Brabender viscosities. Still, the amount of crosslinking agent used for reaction will generally vary from about 0.01% to about 0.07% by weight, depending on the water fluidity of the starch. The exact range can also depend on the pregelatinization process. The type of crosslinking agent used can result in a larger or smaller amount employed. However, in all cases the amount of crosslinking agent must be at least 0.005% by weight.

Any starch or starch blends having suitable properties for use herein may be purified, either before or after any modification or conversion, by any method known in the art to remove starch off flavors, odors, or colors that are native to the starch or created during processing. Suitable purification processes for treating starches are disclosed in the family of patents represented by European Patent No. 554 818. Alkali washing techniques are also useful and described in the family of patents represented by U.S. Patent Nos. 4,477,480 and 5,187,272.

The starch may be used in any amount necessary to achieve the characteristics desired for the particular end use application. In general, the starch is used in an amount of at least up to about 4% by weight of the product.

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Starches suitable for the present invention include amylopectin starches having long outer branches, e.g., waxy corn, where the amylopectin is derived from starch base. Amylose-containing starches from a variety of bases are also useful. These amylopectin and amylose-containing starches can be in their native form or lightly converted. Other useful starches include the above starches that have been enzyme or acid converted to a lower molecular weight. Also useful are amylopectin and amylose-containing starches that have been crosslinked or dextrinized. A light stabilization ($DS < 0.02$) can be tolerated as long as the starch gels under freezer conditions in the ice cream system. Preferably, the starch is an amylopectin or amylose-containing starch wherein the amylopectin is derived from a starch base having long outer branches. Preferably, the starch base is waxy corn.

Also preferred is a starch that gels during freezing. Gels are defined in many different ways in the art. For the purpose of the present invention, the gel character of a particular ice cream mix after holding in the freezer is quantified based on the measurement of the tangent of the phase angle ("tan δ ") for that sample. The tan δ for materials varies from values much higher than one to values much lower than one. Those materials which have tan $\delta > 1$ are typically viewed as liquid-like (under the conditions of measurement) while those with values of tan $\delta < 1$ are viewed as solid or gel-like. Materials which have tan $\delta = 1$ are often described as critical gels, meaning that they represent a critical state of matter between liquids and solids. In the present invention, it has been discovered that those ice cream mixes having starches with a tan $\delta < 1$ after holding in the freezer provide the best resistance to melt-down, and therefore provide the best shape retention of frozen novelties. Accordingly, starches of the present invention that gel during freezing preferably have a tan δ less than about one. More preferably, the starches of the present invention result in a tan δ of less than about 0.4 when measured in a model ice cream system.

In addition to the starch, the ingredients used for making the frozen dessert include milkfat, milk solids-not-fat, sweeteners, stabilizers, emulsifiers and water. The fat component can be vegetable or animal fat, hydrogenated or otherwise. The vegetable fat can be a mixture of fats, such as palm, coconut or palm kernel oil. The milk solids-not-fat can include powdered or concentrated skimmed milk, as well as powdered or concentrated defatted sweet whey.

Sweeteners commonly used include sucrose, glucose, fructose, corn syrup or corn syrup solids having a dextrose equivalent ("DE") varying from about 20 to about 65. The frozen

dessert can include a combination of sweeteners. Ingredients can also include colorings and/or flavorings. Further, the frozen dessert can optionally include fruit or fruit pieces, nuts or candies.

Stabilizing agents such as those previously discussed can be used in the formulation of the frozen dessert. Likewise, emulsifiers such as those previously discussed can be used. The stabilizer(s) and emulsifier(s) are used in an amount of about 0.0% to about 1.0%. Typically, as the amount of fat in the frozen dessert decreases, the amount of stabilizer increases.

The proportion of the above ingredients, such as the ratio of starch and sweeteners, permits increased stability of the products as well as a reduction in the cost of the products. According to the present invention, this stability is made through the replacement of at least a portion of the total solids with the starch. By replacing at least a portion of the solids with the starch, the solid content of the product is maintained while maintaining the smoothness and creaminess of the product. By at least partially replacing the solids with starch, the melt down of the products is slowed and greater heat shock stability provided.

The process for preparing the frozen desserts is as follows. A mixture of the ingredients is prepared, with the mixture blended long enough so as to avoid foaming and ensure proper hydration of the ingredients. The mixture is then pasteurized and homogenized according to standard techniques in the art. The homogenization can be done in one or two steps. The mixture is then cooled to a refrigeration temperature, which is typically about 2°C to about 8°C, and aged as needed, typically for a period of about 4 to about 24 hours. The steps of pasteurization, homogenization and cooling can be done in batch steps or continuously. After aging, the mixture is then rapidly frozen, typically at about -2°C to about -18°C to a product overrun of about 30% to about 120%. During freezing and any subsequent temperature cycling, gelation of the starch can occur. This frozen mixture is then hardened, typically to a temperature of about -20°C to about -40°C for a period of about 12 to about 48 hours.

In the examples that follow, all parts and percentages are given by weight and all temperatures in degrees Centigrade (°C) unless otherwise indicated. The following examples are presented to further illustrate and explain the present invention and should not be taken as limiting in any regard. All percents used are on a weight/weight basis. The following analytical and testing procedures were used to characterize the starch products herein.

EXPERIMENTAL - PROCEDURAL

A. Starches Evaluated

Numerous starches were evaluated for effectiveness as to shape retention (*i.e.*, melt down) and iciness (*i.e.*, formation of ice crystals) in ice cream against a low fat (5%) control and a regular or full fat (10%) commercially available ice cream. Sixteen illustrative starch samples are identified in Table I below –

Table I – Description of Starches

ID #	Short Description	Starch Preparation
1	Native waxy corn starch	unmodified waxy corn starch
2	Acid converted waxy corn starch (20wf)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 20 is obtained
3	Acid converted waxy corn starch (40wf)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 40 is obtained
4	Acid converted waxy corn starch (60wf)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 60 is obtained
5	Acid converted sago starch	sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained
6	Native regular corn starch	unmodified regular corn starch
7	Acid converted sago starch treated with octenyl succinic anhydride	sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained and further treated with OSA to obtain approx 1.75% bound OSA
8	Acid converted waxy corn starch (80wf)	waxy corn starch treated with up to approx 3.0% HCl at 52 C until a desired WF up to 80 is obtained
9	Native waxy rice starch from Bangkok Starch Company	unmodified waxy rice starch
10	Waxy corn starch treated with propylene oxide and phosphorous oxychloride	waxy corn starch treated with PO to obtain approx 6.6% bound PO and further treated with approx 0.004% POCl ₃
11	Tapioca starch treated with propylene oxide and phosphorous oxychloride	Tapioca starch treated with PO to obtain approx 5.0% bound PO and further treated with approx 0.011% POCl ₃
12	Acid converted waxy corn starch treated with propylene oxide	Waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 60 is obtained and further treated with PO to obtain approx 5.2% bound PO
13	Acid converted sago starch treated with propylene oxide	Sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained and further treated with PO to obtain approx 5.4% bound PO
14	Acid converted sago starch treated with sodium tripolyphosphate	Sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained and further treated with STP to obtain approx 0.43% bound PO ₄
15	Tapioca canary pyrodextrin	Tapioca starch treated with anhydrous hydrogen chloride gas to a pH of approx 2.8 and further dry roasted to a maximum temperature of 154 C and held at temperature until approx 100% solubility and approx 2.2 ABF viscosity is obtained

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16	Waxy corn starch enzymatically debranched	dispersed waxy corn starch treated with approx 8% Promozyme 400L (from Novo) until approx 85% short-chain-amylose is obtained and recovered by spray-drying
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B. Ice Cream Preparation

Three separate low fat (5%) ice cream formulations were prepared according to the formulation and procedure defined below –

Table II – Formulation for Low Fat (5%) Ice Cream Mix

Ingredients	Control Formula (%)	2% Starch Formula (%)	4% Starch Formula (%)
Heavy Cream	13.10	13.10	13.10
Skim Milk	60.80	60.80	60.80
Skim Milk Powder (NFDm)	4.66	4.66	4.66
Sucrose	10.00	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.50	3.50
Corn Syrup Solids DE 24	5.50	4.50	3.50
Stabilizer/Emulsifier Blend	0.44	0.44	0.44
Starch	-	2.00	4.00
Total	100.00	100.00	100.00

Forty (40) percent of the total sucrose and the emulsifier/stabilizer blend were dispersed in the skim milk and mixed for five (5) minutes. A dry blend of NFDm, the remaining sucrose, corn syrup solids and the starch product to be evaluated (as required) was added and mixed for ten (10) minutes. The heavy cream was then added to the mixture, and the mixture gently blended so as to avoid foaming. With all ingredients blended together, the mixture was then pasteurized, homogenized and cooled to 4°C, respectively. Pasteurization was performed at 82°C for thirty (30) seconds, and homogenization was done in two stages, first at 17 MPa and then at 3.4 MPa. Flavoring was then added to the ice cream. The ice cream mixture was aged overnight at 4°C. This mixture was then frozen in a continuous freezer (Technogel 100, available from Waukesha Cherry-Burrell, Delavan, Wisconsin) to an overrun of 100% by keeping the flow rate constant and adjusting the air supply. The mixture was filled into eight ounce (237 ml) sample cups, cooled to -20°C, and then hardened at -30°C for 24 hours. The resulting ice cream contains approximately 5% butterfat, 10% milk solids non-fat ("MSNF"), and 37% solids, based on a calculation using typical nutritional data.

C. Heat Shock Stability Test

Temperature fluctuation in the distribution chain was simulated by exposing all ice cream samples to 12 temperature cycles in a freezer (model 34-25 commercially available from ScienTemp Corporation, Adrian, Michigan). The freezer was filled with one layer of 100 samples so that airflow could occur around all samples. Cycling conditions were -18°C for 12 hours followed by -6°C for twelve hours.

D. Melt Down Test

An ice cream sample was placed on a screen in a temperature-controlled cabinet at 19°C. A receptacle was placed on a balance below the screen to collect and weigh the meltdown or drip loss (m_d) over a time period of up to four (4) hours. The initial weight of the ice cream (m_0) was determined and kept within a ten percent (10%) standard deviation. Weight of the drip loss (m_d) was recorded over the four-hour time period. Total melt down (MD, %) was determined at the end of the four-hour period according to the following equation –

$$MD = \frac{m_d}{m_0}$$

E. Sensory Evaluation

Samples were evaluated by an expert panel for 10 different attributes as listed and defined below. During each session, two test samples were rated on a scale of 0-15. A control sample that had gone through temperature cycling was used as a base reference and presented with the samples. Each test sample was evaluated twice to reduce the standard deviation.

Blind testing a control product in five (5) different sessions as a test sample determined reproducibility between the panels. This resulted in a standard deviation of 0.5 for each descriptor, confirming sufficient training of the panel.

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DESCRIPTORS

- Firmness -** Force required to compress the sample with teeth and tongue.
- Coldness -** Tendency to cool surfaces of the mouth.
- Cohesiveness -** Tendency to resist loss of structure during chewing. A cohesive product is elastic and maintains structure during chewing. A non-cohesive product is brittle, easily fragments or is crumbly in texture, and provides a clean bite through.
- Rate of Breakdown -** Rate at which a product softens or fragments upon chewing.
- Ice crystals -** Perception of 'grittiness' caused by the number and size of ice crystals. Ideally, no ice crystals can be detected in the mouth.
- Melt Thickness -** Viscosity of film left on the palate.
- Smoothness -** Tendency of the film on the palate to be smooth, slick and non-sticky.
- Gumminess -** Residual film in the mouth is tacky, sticking the tongue to the surfaces of the mouth.
- Vanilla Flavor -** Authentic not of vanilla and cream.
- Off-Note -** Any inappropriate aroma.

F. Rheological Testing Procedure

Each of the ice cream mixes was poured into molds and held in the freezer at -18°C . One mold of each formulation was removed from the freezer and tested on the rheometer as soon as it equilibrated to 25°C . Another mold was removed from the freezer and placed in a constant temperature bath at -6.7°C where it was held for 7-8 days, before it was removed, equilibrated to 25°C and tested on the rheometer.

A sequence of rheology experiments was performed on each sample as described in Table III below -

Table III - Rheology Tests Run on Each Sample

Test Name	Specific Conditions
Dynamic Strain Sweep	$\omega=1\text{rad/s}$, $\gamma_{\text{min}}=1\%$, $\gamma_{\text{max}}=100\%$
Dynamic Frequency Sweep	$\gamma < \gamma_{\text{cr}}$, $\omega_{\text{min}}=1\text{rads}^{-1}$, $\omega_{\text{max}}=100\text{rads}^{-1}$
Steady Shear Step Rate	Shear rate= 1s^{-1} , length of experiment= 120 s
Steady Shear Rate Sweep	Time per shear rate= 30s , shear rate range= $1\text{-}100\text{s}^{-1}$

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In Table II, "min" refers to minimum, "max" refers to maximum, " ω " refers to frequency, " γ " refers to strain, "rad" refers to radians, and "s" refers to seconds. Rheological tests described above are according to standard rheological terms.

Experiments on the ice cream mixes in the molds were all done at 25°C. The data tabulated from these experiments includes: G' at 10 rad/s, $\tan \delta$ at 10 rad/s, γ_{cr} , the peak in steady shear viscosity, η at 1/s, the final η after 120s at 1/s, and η at 100/s.

The melt down test used is described above in Section C. The procedure involves placing a known mass of the frozen ice cream on a wire mesh and then recording the weight loss over time. For the current study, the percent weight loss of the ice cream after 4 hours at 19°C was used.

G. Differential Scanning Calorimetry Procedure for Determining T_g'

Differential scanning calorimetry measurements were performed in a Perkin-Elmer DSC-7 with CCA cooling system (Norwalk, Connecticut). Liquid N_2 was used for sub-ambient temperature experiment. The instrument was calibrated with indium and water. An empty stainless-steel pan was used as a reference.

Approximately 10 mg of starch (dry base) was weighed into large volume stainless steel pan. Extra water was added to the sample to reach the desired moisture content. Each starch was tested at two solid levels (40% and 20%) using quench cooling and annealing method to ensure that the measured T_g' and W_g' values are close to real values. The starch/water mixture was first heated from 10°C to 160°C at 10°C/min to ensure that all the starch was fully dispersed in water. Then the starch/water mixture was quench cooled to -70°C and was held for 15 minutes to ensure maximum amount of ice formed. The sample was then heated at 2.5°C/min. to detect the T_g' and the amount of the frozen water. To maximize the amount of ice formed in the ice cream mix, the samples were also subjected to an annealing procedure before temperature scanning. The sample was held at the temperature slightly above T_g' for 30 minutes and then was cooled to below T_g' at 1.0°C/min and held at -40°C for another 10 minutes. The samples were then heated at 2.5°C/min to 20°C. The middle point of the glass transition was reported as T_g' , and the ice melting enthalpy was used to calculate the W_g' value of the starch. Duplicate tests were run for each solid level or cooling condition. The average values from those tests that generated maximum amount of ice were reported.

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EXPERIMENTAL - RESULTS

1. Melt Down and Sensory Results

The effect of the starch according to the present invention on the melt down on ice cream is shown in Figure 1. From Figure 1 it is seen that unmodified or lightly crosslinked starches are preferred. From Figure 1 it is also seen that waxy starches are preferred as the base starch, more preferably, waxy corn starches.

Table IV - Meltdown/Ice Crystal Data on Ice Creams with Starch

Sample ID	Meltdown	Ice Crystals
Starch 16 at 2%	43.00	5.85
Starch 16 at 4%	23.93	4.45
Starch 2 at 2%	16.00	4.7
Starch 3 at 2%	21.00	4.85
Starch 4 at 2%	37.00	4.75
Starch 4 at 4%	4	2.85
Starch 8 at 2%	44.00	4.8
Control ice cream without starch	59	7.5
Starch 12 at 2%	79.00	6.45
Starch 12 at 4%	97.00	3.45
Starch 14 at 2%	25.00	4.8
Starch 13 at 2%	68.00	5.15
Starch 7 at 2%	49.00	4.85
Starch 1 at 2%	14.37	4.7
Starch 6 at 2%	19.73	5.45
Starch 9 at 2%	88.18	5.36
Starch 10 at 2%	80.09	5.1
Starch 5 at 2%	37.10	6.7

The correlation between the reduced melt down of ice cream and the ability of the starch to gel in the model ice cream system under freezer conditions is shown in Figure 2. As can be seen from Figure 2, a starch having a $\tan \delta$ of less than about 1.0 results in a frozen dessert having a melt down of about fifty percent (50%) or less. Starches having a $\tan \delta$ of less than about 0.4 provided the best performance, resulting in an ice cream with the slowest melt down.

As previously noted, the results of the effectiveness of the above samples with respect to melt down are illustrated in Figure 1. Those frozen desserts containing about two to about four percent of a starch that gels during freezing, particularly those starches having a $\tan \delta$ less than about one, resulted in reduced melt down and improved shape retention after exposure to heat shock.

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Those skilled in the art recognize that certain processing conditions (e.g., reduced overrun) and variations in the formula (e.g., higher fat content) can result in improved melt down. The present invention shows that the addition of certain starches improves meltdown, even more than presently used emulsions and stabilizers.

2. Rheological Testing Results

The ice cream mixes show significant changes in rheology during storage at -6.7°C . For most of the samples, $\tan \delta$ decreases over time, which indicates the development of gelled structures under frozen conditions. Trends are similar for low-fat and non-fat formulas. After 7-10 days, the least gelled (highest $\tan \delta$) samples are Sample 16 and Sample 12 while the most gelled (lowest $\tan \delta$) samples are Sample 1 and Sample 3. The viscosity of each mix increased or stayed constant throughout the time that the samples were held at -6.7°C . The modulus, G' , showed similar behavior to the viscosity.

Table V - Rheology Data on Ice Cream Mixes with Starch

Sample	$\tan \delta$ at 10 rad/s fresh	η at 1/s fresh	G' at 10 rad/s fresh	$\tan \delta$ at 10 rad/s @ 7-8 days	η at 1/s 7-8 days	G' at 10 rad/s 7-8 days
Starch 16 at 2%	1.5724	0.2476	0.7279	0.8978	0.9230	3.2386
Starch 16 at 4%	1.2644	0.4205	1.4463	0.472	3.8639	22.018
Starch 2 at 2%	1.6801	0.3120	1.6682	0.2538	15.2720	193.6
Starch 3 at 2%	2.0387	0.2614	0.9887	0.3665	7.9273	52.157
Starch 4 at 2%	2.2618	0.2089	0.6088	0.3597	6.4949	48.769
Starch 4 at 4%	1.8449	0.3314	1.5129	0.3263	46.5130	865.84
Starch 8 at 2%	1.9657	0.2037	0.5129	0.7069	2.8037	12.249
Control ice cream mix without starch	1.7367	0.1982	0.5058	1.6172	0.3039	0.9667
Starch 12 at 2%	2.2580	0.2277	0.686	1.93705	0.2699	0.9439
Starch 12 at 4%	2.0553	0.3717	1.4746	1.6779	0.5526	2.5249
Starch 14 at 2%	0.9583	1.2793	3.9336	0.36985	12.4740	91.049
Starch 13 at 2%	2.1291	0.2271	0.6886	1.3858	0.5244	1.9084
Starch 7 at 2%	1.3449	0.5992	1.7108	0.46385	4.7607	20.386
Starch 1 at 2%	1.9E+00	5.1E-01	2.0E+00	0.31	1.2E+01	8.8E+01
Starch 6 at 2%	7.2E-01	2.6E+00	9.5E+00	0.31	1.8E+01	1.3E+02
Starch 9 at 2%	2.1E+00	4.4E-01	1.8E+00	1.4	7.6E-01	3.1E+00
Starch 10 at 2%	2.1E+00	7.0E-01	2.5E+00	1.5	9.2E-01	4.2E+00
Starch 5 at 2%	1.5E+00	5.2E-01	1.5E+00	0.6	1.8E+00	5.8E+00

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3. T_g' Results

Those starches identified as providing good ice crystal growth inhibition capability included native starches, acid or enzyme converted starches, or chemically modified versions of those starches having a DE less than about 8, and a T_g' greater than about -6°C (T_g' > ~ -6°C) and, water binding property of Wg' greater than about 0.30 (Wg' > ~ 0.30) (unfreezable water weight fraction in the unfrozen glass state). T_g' and Wg' are determined by the calorimetry method described above. The results are provided in Table VI below –

Table VI

Starch Sample	T _g ' (°C)	Wg'(g/g total glass)
Starch 1	-4.521	0.309
Starch 4	-5.627	0.352
Starch 8	-5.321	0.325
Starch 11	-5.993	0.308
Starch 7	-5.127	0.310
Starch 14	-5.550	0.318
Starch 12	-7.766	0.322
Starch 13	-8.370	0.257
Starch 16	-7.021	0.300
Starch 15	-8.651	0.269

In the present invention, corn syrup solids were reduced to keep the total solids content of the formulation constant. Adding starch to replace fat can affect the glass transition temperature, freezing temperature and the 'water binding' property of the serum phase in the frozen state. Starches were compared at 2% and 4% usage levels. At about 3% to about 4% usage level, the starches of the present invention function in the absence of other stabilizers, providing improved heat shock stability characterized by a reduction in ice crystals as compared to a non-starch control. Higher levels of starch generally further improve performance with respect to melt down and heat shock stability. However, above about 4%, negative textural attributes occur.

4. Low Fat Ice Cream with Starch

Ice cream formulations were prepared according to the formulation defined in Table VI below –

Table VI - Ice cream formulation 5% butterfat, 3% starch #4, no other stabilizer:

Ingredients	Control Formula (%)	3% Starch Formula (%)
Heavy Cream	13.10	13.10
Skim Milk	60.80	60.80
Skim Milk Powder (NFDN)	4.88	4.88
Sucrose	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.15
Corn Syrup Solids DE 24	5.50	4.15
Emulsifier*	0.44	0.14
Starch 4	-	3.00
Total	100.00	100.00

* The emulsifier for the control is the stabilizer/emulsifier blend provided above. The emulsifier in the starch formula has the stabilizer removed.

As in the 'Ice Cream Preparation' section above, when starches of the present invention were added, the 42DE corn syrup solids and the 24DE corn syrup solids were reduced to keep the total solids constant. However, here the stabilizer was also omitted. Each ice cream mix was prepared according to the procedure described in the 'Ice Cream Preparation' section.

H. Non-fat Ice Cream Mix with Starch and Stabilizer

Ice cream formulations were prepared according to the formulation defined in Table VII below --

Table VII - Rheological Ice Cream Formulation

Ingredients	1
	0% Fat
Heavy Cream	0.00
Skim Milk	72.67
Non-Fat Dry Milk	4.82
Sucrose	10.50
42DE Corn Syrup Solids	5.78
24DE Corn Syrup Solids	5.78
CREST Stabilizer/Emulsifier	0.46

As in the 'Ice Cream Preparation' section above, when starches of the present invention were added, the 42DE corn syrup solids and the 24DE corn syrup solids were reduced equally to keep the total solids constant. Each ice cream mix was prepared according to the procedure described in the 'Ice Cream Preparation' section. Seven starches were evaluated in the non-fat formula at 2.1% wt/wt.

The ice cream mixes show significant changes in rheology during storage at -6.7°C . For most of the samples, $\tan \delta$ decreases over time, which indicates the development of gelled structures under frozen conditions. After 7-10 days, the least gelled (highest $\tan \delta$) samples are Sample 16 and Sample 12 while the most gelled (lowest $\tan \delta$) samples are Sample 1 and Sample 3. The viscosity of each mix increased or stayed constant throughout the time that the samples were held at -6.7°C . The modulus, G' , showed similar behavior to the viscosity.

Table IX -- Rheology Data on Non-fat Ice Cream Mixes

Sample	$\tan \delta$ at 10 rad/s fresh	η at 1/s fresh	G' at 10 rad/s fresh	$\tan \delta$ at 10 rad/s @ 7-8 days	η at 1/s 7-8 days	G' at 10 rad/s 7-8 days
Starch 1 at 2.1%	3.9E+00	2.1E-01	4.4E-01	2.6E-01	1.5E+01	1.0E+02
Starch 6 at 2.1%	9.0E-01	1.3E+00	4.2E+00	3.1E-01	8.0E+00	7.4E+01
Starch 10 at 2.1%	3.1E+00	3.2E-01	7.8E-01	2.1E+00	5.0E-01	1.5E+00
Starch 4 at 2.1%	4.0E+00	5.8E-02	1.2E-01	3.8E-01	7.8E+00	4.6E+01
Starch 5 at 2.1%	1.5E+00	4.1E-01	8.4E-01	3.9E-01	2.6E+00	1.4E+01

Although the present invention has been described and illustrated in detail, it is to be understood that the same is by way of illustration and example only, and is not to be taken as a limitation. The spirit and scope of the present invention are to be limited only by the terms of any claims presented hereafter.

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What is claimed and desired to be secured by Letters Patent is:

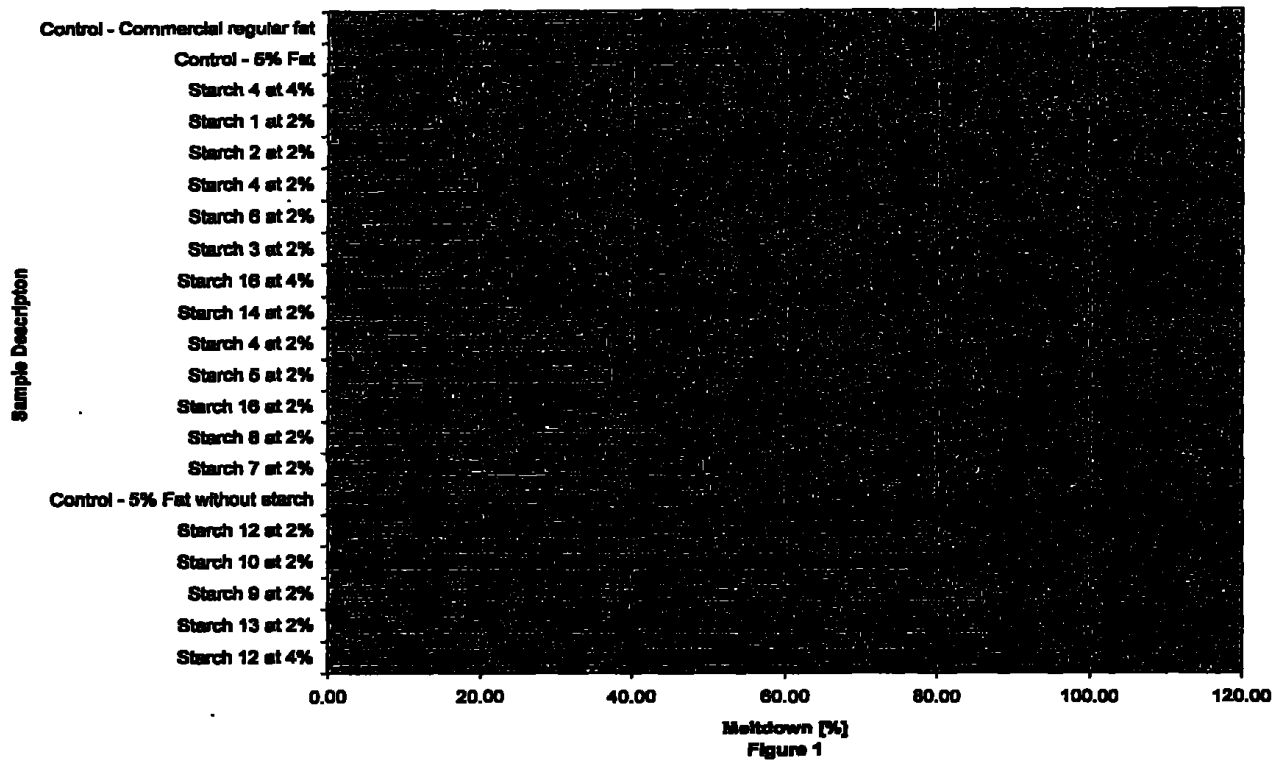
- 1. A starch for use in frozen desserts, the starch comprising a $\tan \delta$ of about 1.0 or less, wherein the starch is able to gel during freezing conditions.**
- 2. The starch of claim 1, wherein the starch is used as a sweetener substitute.**
- 3. The starch of claim 1 further comprising a $\tan \delta$ of about 0.4 or less.**
- 4. The starch of claim 1 wherein the starch has been converted to a water fluidity ("WF") of from about 0 to about 80.**
- 5. The starch of claim 4 wherein the starch has been further converted to a WF of from about 20 to about 60.**
- 6. The starch of claim 1 wherein the starch is an amylopectin starch.**
- 7. The starch of claim 6 wherein the amylopectin starch is waxy corn.**
- 8. The starch of claim 1 wherein the starch provides a reduction in the melt down of the frozen dessert of at least 100% versus frozen desserts without the starch.**
- 9. The starch of claim 1 wherein the starch has a glass transition temperature T_g' of about -6°C or greater, a water binding property Wg' of about 0.30 g/g or greater, and is thereby able to provide improved ice crystal inhibition in a frozen dessert versus a frozen dessert without the starch.**

10. A process for preparing a frozen confectionery, the process comprising the steps of:
- preparing a mixture of ingredients for the frozen confectionery, the mixture including a starch having a $\tan \delta$ of about 1.0 or less,
 - pasteurizing the mixture within a temperature range of from about 80°C to about 90°C;
 - homogenizing the mixture within a pressure range of from about 2MPa to about 20 MPa;
 - cooling the mixture within a temperature range of from about 2°C to about 8°C;
 - aging the mixture for a time period of about 4 hours to about 24 hours;
 - freezing the mixture within a temperature range of from about -2°C to about -8°C; and
 - hardening the mixture at a temperature of from about -20°C to about -40°C;
- wherein the step of freezing effects gelatinization of the starch, and
- wherein the frozen confectionery has an improvement in melt down of at least 100% over frozen confectionery that do not have the starch as an ingredient.
11. The process according to claim 10 wherein the starch is an amylopectin starch.
12. The process according to claim 11 wherein the amylopectin starch is a waxy corn.
13. The process according to claim 10 further comprising the step of replacing at least part of sweetener ingredients of the frozen confectionery with the starch.
14. A frozen dessert comprising a starch having a $\tan \delta$ of about 1.0 or less, wherein the starch gels during freezing conditions.

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ABSTRACT

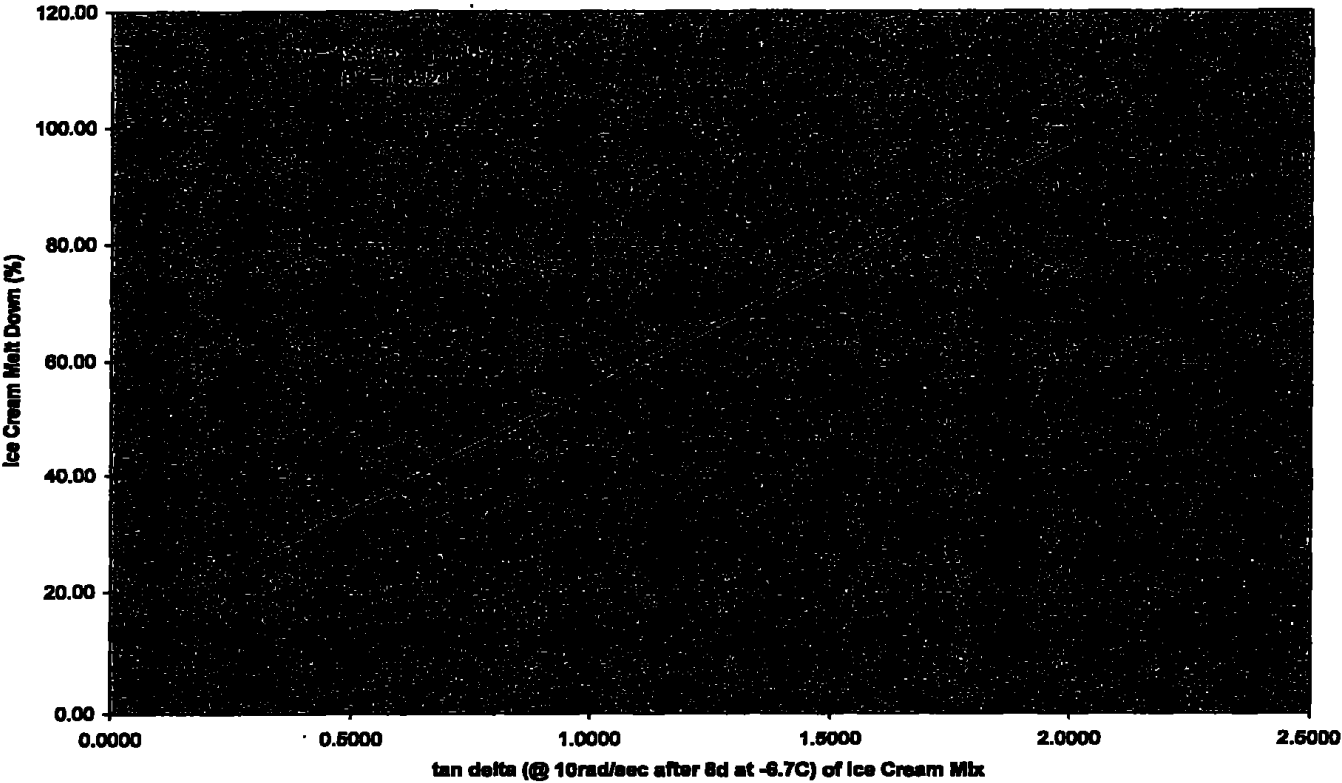
Use of starch and starch derivatives in frozen desserts for improved melt down and shape retention. These improved characteristics are retained in the frozen dessert even after multiple heat shock cycling.



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Figure 2

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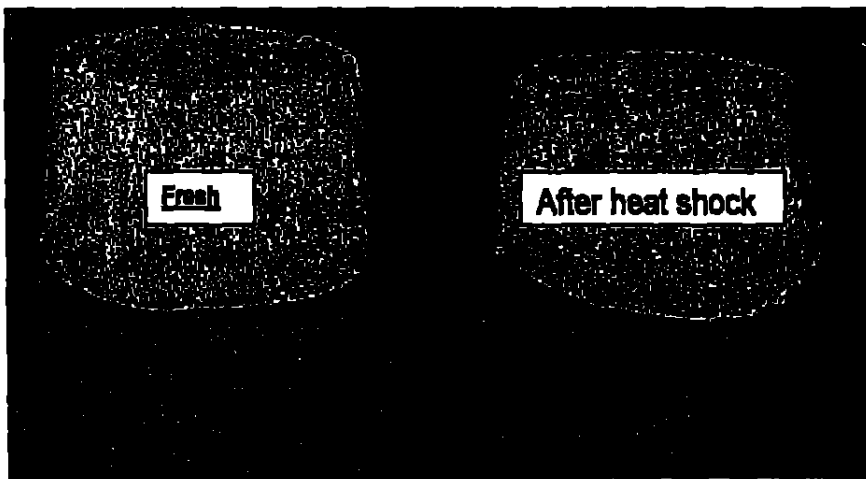


Figure 3. Photo of fresh and heat shocked ice cream containing 3% starch

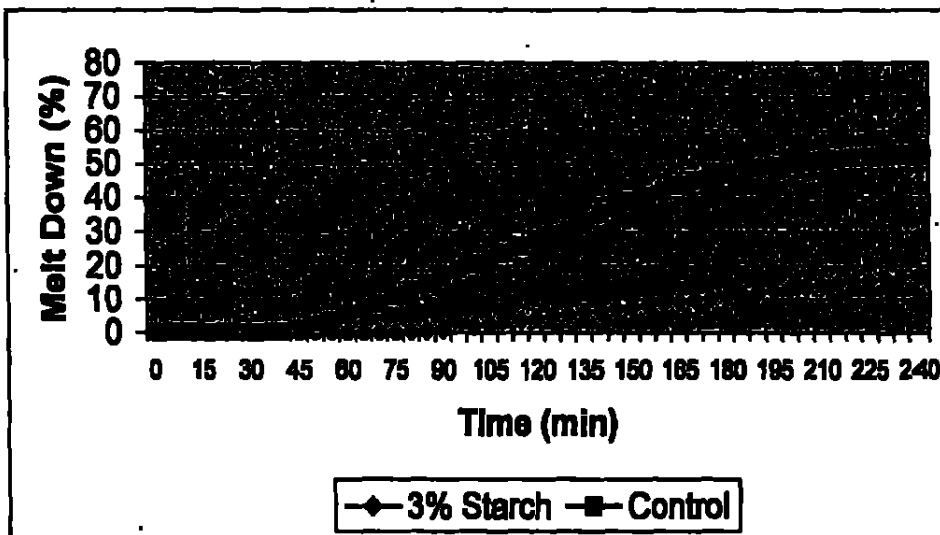


Figure 4. Melt curve of ice cream containing 3% starch

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FECHA DE PRESENTACIÓN: **Octubre 16, 2003**

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JUAN PABLO PIEDRAHITA AGUILERA
TRADUCTOR E INTERPRETE OFICIAL
RES. 718 DE MAYO 2003
DEL MIN. INTERIOR Y DE JUSTICIA

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Lo que se reivindica y para lo cual se desea obtener protección mediante el Título de Patente es lo siguiente:

1. Un almidón para uso en postres congelados, el almidón que comprende una δ "tan" de 1.00 o menor, donde el almidón es capaz de convertirse en gel durante las condiciones de congelamiento.
2. El almidón de la reivindicación 1, en el cual el almidón se utiliza como endulzante sustituto.
3. El almidón de la reivindicación 1, que además comprende una δ "tan" de 0.4 o menor.
4. El almidón de la reivindicación 1, donde el almidón se ha convertido a una fluidez de agua ("FA") de cerca de 0 a cerca de 80.
5. El almidón de la reivindicación 1, donde el almidón se ha convertido posteriormente a una FA de cerca de 20 a cerca de 60.
6. El almidón de la reivindicación 1, donde el almidón es un almidón amilopectina.
7. El almidón de la reivindicación 1, donde el almidón amilopectina es un maíz ceroso.
8. El almidón de la reivindicación 1, donde el almidón suministra una reducción del derretimiento del postre congelado de por lo menos 100% comparado a postres congelados sin el almidón.
9. El almidón de la reivindicación 1, donde el almidón tiene una temperatura de transición vítrea (T_g ') de cerca de -6° C o mayor, o una propiedad ligante

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de agua (W_g ') de cerca de 0.30 g/g o mayor, y por ende es capaz de suministrar inhibición mejorada de cristales de hielo en un postre congelado comparado con un postre congelado preparado sin el almidón.

10. Un proceso para preparar confitería congelada, dicho proceso comprendiendo los siguientes pasos:

preparación de una mezcla de ingredientes para la confitería congelada, la mezcla incluyendo un almidón con una δ "tan" de 1.00 o menor,

pasteurizando la mezcla dentro de un rango de temperatura de cerca de 80°C a cerca de 90°C;

homogenizando la mezcla dentro de un rango de presión de cerca 2Mpa a cerca de 20Mpa;

enfriando la mezcla en un rango de temperatura de cerca de 2°C a cerca de 8°C;

añejando la mezcla durante un periodo de tiempo de cerca de 4 horas a cerca de 24 horas;

congelando la mezcla dentro de un rango de temperatura de cerca de -2°C a cerca de -8°C; y endureciendo la mezcla a una temperatura de cerca de -20°C a cerca de -40°C;

donde el paso de los efectos de congelamiento y conversión en gel del almidón y donde la confitería congelada muestra un derretimiento mejorado de por lo menos 100% comparada con confitería congelada que no tiene el almidón como ingrediente.

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11. El proceso, de acuerdo con la reivindicación 10 donde el almidón es un almidón amilopectina.

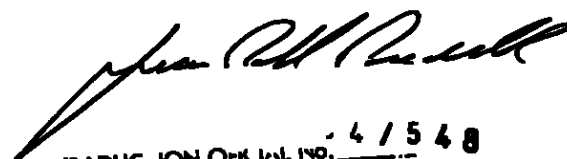
12. El proceso, de acuerdo con la reivindicación 11, donde el almidón amilopectina es un maíz ceroso.

13. El proceso, de acuerdo con la reivindicación 10 que además comprende el paso de reemplazar por lo menos parte de los ingredientes endulzantes de la confitería congelada con el almidón.

14. Un postre congelado que comprende un algodón con una δ "tan" de 1.00 o menor, donde el almidón es capaz de convertirse en gel durante las condiciones de congelamiento.

ES TRADUCCIÓN FIEL Y COMPLETA de la cual se deja copia en los archivos de esta oficina para su confrontación y a la cual me remito.

Bogotá, Noviembre 29, 2004.


TRADUCCION OFICIAL No. 4 / 5 4 8
JUAN PABLO PIEDRA
TRADUCTOR E INTERPRETE OFICIAL
RES. 718 DE MAYO 2000
DEL MIN. INTERIOR Y DE JUSTICIA



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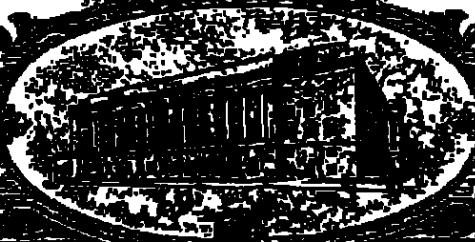
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Item		No de unidades	
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2	DVD		
3	REVISTA		
4	LIBRO		
5	PERIODICO		
6	DISQUETES		
7	SOBRE DE MANILA		
8	SOBRE BLANCO TAMAÑO CARTA		
9	SOBRE BLANCO TAMAÑO OFICIO		
10	OTROS:		

Observaciones:

Arte final

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APPLICATION NUMBER: 60/498,834
FILING DATE: August 29, 2003

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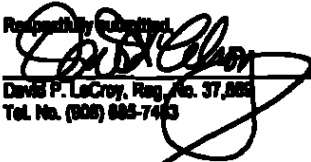
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PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53 (c)(2).

INVENTOR(s)/APPLICANT(s)					
LAST NAME		FIRST NAME		MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)
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TITLE OF THE INVENTION (280 characters max)					
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STATE	New Jersey	ZIP CODE	08807	COUNTRY	U.S.A.
ENCLOSED APPLICATION PARTS (check all that apply)					
☒ Specification (and any claims)		Number of Pages: 22		☒ Abstract Number of pages: 1	
☒ Drawing(s)		Number of Sheets: 3		☐ Other (specify)	
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The Commissioner is hereby authorized to charge filing fees and additional fees required to Deposit Account Number: 14-0488.				PROVISIONAL FILING FEE AMOUNT (\$): 160.00	

- ☒ The invention was not made by an agency of the United States Government or under a contract with an agency of the United States Government
- ☐ A Power of Attorney accompanies this cover sheet.

Respectfully submitted,

David P. LeCroy, Reg. No. 37,865
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Date: August 28, 2003

- ☐ Additional inventors are being named on separately numbered sheets attached hereto.

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STARCH FOR FROZEN DESSERTS

BACKGROUND OF THE INVENTION

Technical Field. The present invention relates to a starch for use in frozen food products. More specifically, the present invention is directed towards a starch or starch derivative for use in frozen desserts in order to obtain improved structure characteristics, including slow melt down and excellent shape retention.

Background Information. In addition to their rich flavor, frozen confectioneries are enjoyed for their creaminess and smoothness. However, in order to preserve these characteristics, these products have to be handled and stored with care. Unfortunately, even small temperature variations can occur during storage, distribution and handling. For example, such variations can occur when a consumer buys a frozen product and does not consume it right away, as in the time from when a consumer purchases such a product at a grocer and places that product in his freezer. Partial or even substantial defrosting of the product can occur before it is refrozen. This cycling of temperature variations can result in ice crystal growth in the product. Such growth affects both the visual appearance and organoleptic properties of the frozen product, thereby reducing its quality and appeal, at least as perceived by the consumer. The most frequently occurring textural defect in ice cream is due to ice crystal growth, and is the primary limitation to its shelf life. As such, it is desirable for frozen desserts to be stable against 'heat shock', or the cyclic conditions of partial thawing and refreezing that occur during typical storage, shipping and handling of these products.

As used herein, a frozen dessert, frozen confectionery or frozen product refers to a product in which water is present in both the liquid and frozen state. Examples of frozen deserts include ice cream, ice milk and parfaits. As used herein, frozen novelties refer to frozen desserts such as those available on a stick or in sandwiches or cones.

Ice crystals in frozen products need to be numerous and of small, nearly uniform size to avoid detection when eaten. Because ice crystals are relatively unstable, during storage they undergo changes in number, size and shape, which is collectively referred to as recrystallization. While some recrystallization occurs naturally at constant temperatures, by far the majority of it occurs due to temperature fluctuations or heat shock. An increase in temperature during the frozen storage of a product such as ice cream causes some of the ice crystals, particularly the

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smaller ones, to melt. As the temperature decreases, that water refreezes without renucleation, instead depositing on the surface of larger crystals. As such, heat shock or temperature cycling in frozen products reduces the total number of ice crystals and increases the mean ice crystal size.

To a consumer, the perception of coldness and iciness of ice cream is directly related to the amount of ice formed in the ice cream and the size of the ice crystals. Typically, consumers can detect ice crystals larger than about fifty (50) microns. Such large ice crystals contribute to the coarseness and iciness of the ice cream. For a typical ice cream, the amount of ice formed and the crystal size distribution are mainly determined by the formulation, processing, and storage conditions as outlined below.

Ice crystallization is also affected by the glass transition temperature (T_g') and the freezing point of the ice cream. In a maximally freeze-concentrated solution, an invariant condition occurs at temperature T_g' and concentration C_g' (or unfreezable water W_g'), depending on the particular mixture of compounds in the formulation. The unfreezable water W_g' is that water rendered unfreezable in the maximum freeze-concentrated solution due to the low diffusivity in the glassy state. When ice cream is stored below T_g' , the system is in the glassy state. In this glassy state, the diffusion-controlled processes that typically result in reduced quality and stability can be largely inhibited. Above T_g' , the system is in a rubbery state where the translational diffusion is free to occur. The rate of diffusion-controlled deterioration increases exponentially with increasing ΔT , the temperature difference between storage temperature T and T_g' ($\Delta T = T - T_g'$), in agreement with WLF kinetics. Practically speaking, the higher the W_g' , the less water there is available for freezing at the same solid content. For frozen desserts such as ice cream, a higher W_g' means less iciness.

Various stabilizing gums have been used as additives in an attempt to improve the heat shock stability and creaminess of frozen food products. Such stabilizing gums have included gelatin, agar, gum acacia, guar gum, carob or guar seed flour, locust bean gum, carrageenan, alginate, carboxymethyl cellulose, xanthan and the like, with each exhibiting their own set of advantages and disadvantages. Microcrystalline cellulose ("MCC") and carboxymethyl cellulose are often used in combination with other stabilizing gums to improve functional effectiveness. MCC and cellulose gum can be used together to reduce ice crystal growth. However, MCC can

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continue to activate during aging or subsequent processing, resulting in unanticipated viscosity. MCC can also be costly and prevent frozen products from carrying a 'natural' label.

While gums improve stability, their use has several disadvantages. For example, the amount of stabilizing gum required to provide heat shock stability can result in a product having an unacceptably greasy and/or gummy mouthfeel. Further, products containing such stabilizing compounds are regulated and often poorly perceived by the public. In addition, these additives can be expensive. Several attempts have been made to improve heat shock stability of frozen products by reducing the amount of stabilizer(s) in the product. However, stabilizers have the added benefit of reducing the size of ice crystals in those products. By replacing all or part of those stabilizers, the size of ice crystals in the product can increase, resulting in a less desirable product.

Emulsifiers provide stability and creaminess to frozen products by facilitating an interface between the aqueous phase and the fat phase of the product. Milk or milk proteins and eggs yolks are natural sources of emulsifiers useful for their water-binding properties. Commercially available emulsifiers are generally derived by chemical reactions with naturally occurring glycerides.

Emulsifiers added to ice cream reduce the stability of this fat emulsion by replacing proteins on the surface of the milkfat. For example, ice cream is a frozen dessert that is both an emulsion and foam. When an ice cream mixture is whipped, the fat emulsion begins to break down and the fat globules begin to flocculate or destabilize. This partially coalesced fat stabilizes air bubbles that are whipped into this mix. Without emulsifiers, the fat globules would be able to resist this coalescing due to the proteins being absorbed to the fat globule, causing the air bubbles to not be properly stabilized and affecting the texture or smoothness of the ice cream.

Emulsifiers also affect the melt down of the ice cream. When ice cream is placed in an ambient environment, the ice cream melts and the fat-stabilized foam structure collapses. Melting is controlled by outside temperature and rate of heat transfer. However, even after the ice crystals melt, the ice cream does not collapse until the fat-stabilized foam structure collapses. This collapse is a function of the extent of fat destabilization/partial coalescence, which is controlled by the emulsifier concentration.

The frozen product ice cream is basically composed of milkfat, serum solids or milk solids-not-fat, sweeteners, stabilizers and emulsifiers, and water. Stabilizers and emulsifiers

were previously discussed. Water found in ice cream comes from the milk or other ingredients. The milkfat or butterfat is typically obtained from sweet or heavy cream, and provides flavor, smooth texture and body to the ice cream. Other types of fats, such as palm oil, can also be used. The serum solids contain the lactose, caseins, whey proteins, minerals and ash content, and can be found in concentrated skimmed milk or skim milk powder. Sucrose is typically the main sweetener. At least a portion of the sweetener can be obtained from corn syrup solids, which provides a firmer and chewier body to the ice cream. Liquid and solid corn syrup is available in various dextrose equivalents ('DE'), which is a measure of the reducing sugar content of the syrup calculated as dextrose and expressed as a percentage of the total dry weight. As the DE increases, the sweetness increases and the molecular weight decreases.

The formulation used for manufacturing ice cream varies depending upon the type of product desired. Types of ice cream products include economy brands typically having the legal minimum fat and total solids content, standard brands, premium brands and super premium brands, as well as a hard frozen, soft frozen, low fat, light, sherbet, sorbet or frozen yogurt product. As the amount of fat increases in the formulation, the ice cream becomes less viscous.

Ice cream ingredients are blended together according to the formulation selected to produce the ice cream mix. The mix is then pasteurized, followed by homogenization. Once homogenized, the mix is aged to allow the fat to cool and crystallize, and the proteins and polysaccharides to fully hydrate. After aging, the mix is frozen, during which particulates such as fruit, nuts, candy, cookies, etc. are added to the mix, followed by hardening of the ice cream, typically at temperatures of -30 to -40°C. The freezing and hardening is best done at a fast rate, as a fast freezing rate promotes the formation of many small ice crystals instead of fewer larger ice crystals.

The temperature at which ice cream freezes is affected or depressed by the concentration of sweeteners and/or their molecular weight. For example, a mix having a higher concentration of sugars and/or sugars of a lower molecular weight will have a lower freezing temperature curve than a mix having a lower concentration of sugars and/or sugars of a higher molecular weight. The lower the freezing curve, the softer the ice cream, the greater the freeze out of the water during hardening, and the greater the susceptibility of the ice cream to heat shock.

For low fat or reduced fat frozen desserts, particularly frozen novelties, shape retention is necessary for product quality. It is known that melt characteristics of frozen desserts are relevant

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to shape retention. For ice cream, melt down is strongly influenced by the fat and air domains of the ice cream. As explained above, both fat and emulsifiers in the dessert affect melt down. For a low or reduced fat formula, melt down can be critical. Simply adding more emulsifier to make up for the loss of fat can be expensive and affect the characteristics of the ice cream, and is therefore not a preferred solution.

Various starch-based products have been marketed and sold for use in frozen desserts. These include converted starches, stabilized starches and crosslinked starches. It is well known in the food industry to use starch as a thickener and/or binder, i.e., as a stabilizer. Typically, starches have been used in frozen confectioneries as a fat substitute or texturing agent. With the trend toward diminishing sugar in frozen products such as ice cream, starch-based products such as hydrogenated starch hydrolyzates (polyols) have proven useful alternatives to sweeteners such as maltodextrin and polydextrose. These starch sweetener substitutes are typically the liquid and/or solid corn syrups previously discussed. However, these substitutes do not function in reducing melt down and/or reducing iciness in frozen desserts.

Accordingly, there is a need for a starch product for use in frozen desserts that reduces the melt down of the dessert when exposed to temperature fluctuations during storage and distribution. Further, there is a need for a starch product for use in frozen desserts that aids in retaining the shape of the frozen product, particularly low fat products. Finally, there is a need for a starch product for use in frozen desserts that reduces the risk of ice crystal formation during heat cycling.

SUMMARY OF THE INVENTION

The present invention is directed towards starch and/or starch derivatives useful in frozen desserts for obtaining improved structure characteristics, including a reduction in melt down, improved shape retention and/or decrease in ice crystal formation during heat cycling. The invention is also directed towards a frozen dessert that includes those starch and/or starch derivatives. The frozen dessert can include fat, sweeteners, milk solids-not-fat, water and starch. The starch and/or starch derivative of the present invention can be used to replace at least a portion of the sweetener.

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BRIEF DESCRIPTION OF THE DRAWINGS

The manner in which these objectives and other desirable characteristics can be obtained is explained in the following description and attached drawings in which:

Figure 1 is a graph illustrating the percent melt down of various ice creams after four hours at 19°C.

Figure 2 is a graph illustrating the correlation between the melt down of ice cream and the tan δ of the mix after eight days at -6.7°C.

Figure 3 is a photograph illustrating the effect in melt down of an ice cream containing a starch of the present invention after heat shock treatment as compared to freshly prepared ice cream.

Figure 4 is a melt down curve of an ice cream containing 3% of a starch of the present invention as compared to a control ice cream containing no starch.

DETAILED DESCRIPTION OF THE INVENTION

The starch base material used for the present invention may be derived from any source, including cereal or root starches. Typical sources for the starches are cereals, tubers, roots, legumes and fruits. Native starch sources include, for example, any variety of corn (maize), pea, potato, sweet potato, banana, barley, wheat, rice, oat, sago, amaranth, tapioca, arrowroot, canna, sorghum and waxy and high amylose varieties thereof. As used herein, "waxy" includes starches containing no more than about 10% amylose by weight. As used herein, the term "high amylose" includes starches containing at least about 40% by weight amylose. As used herein, the term "amylose-containing" includes those starches containing at least about 10% by weight amylose. Preferably, the starch base material is an amylopectin starch such as a waxy corn starch or amylose-containing starch.

The granular starch base can be one that has been lightly converted or hydrolyzed to a water fluidity ("WF") of about 0 to about 80. The term "water fluidity" has a very specific meaning as described further herein below. The starch is converted to its fluidity or thin-boiling form using a suitable method of degradation that results in the modified starch defined herein. Such degradation includes, for example, mild acid hydrolysis with an acid such as sulfuric or hydrochloric acid, conversion with hydrogen peroxide, or enzyme conversion. Converted starch products can include blends of different starches converted by various techniques as well as

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converted starch(es) blended with unconverted starch(es). Commercially, starch is typically converted by acid or enzyme conversion techniques. Hydrogen peroxide can also be used to convert or thin the starch, either alone or with metal catalysts.

As noted above, the starch of the present invention can be converted to a water fluidity ("WF") of from about 0 to about 80, particularly from about 20 to about 60. Water fluidity, as used herein, is an empirical test of viscosity measured on a scale of 0-90 wherein fluidity is inversely proportional to viscosity. Water fluidity of starches is typically measured using a rotational shear-type viscometer (commercially available from Thomas Scientific, Swedesboro, New Jersey), standardized at 30°C with standard oil that has a viscosity of 24.73 cps and requires 23.12 ± 0.05 sec for 100 revolutions. Accurate and reproducible measurements of water fluidity are obtained by determining the time which elapses for 100 revolutions at different solids levels depending on the starch's degree of conversion – as the degree of conversion increases, the viscosity decreases and the WF values increase.

The base material can be modified either chemically or physically using techniques known in the art. The modification can be to the base or the converted starch, though typically the modification is carried out after conversion.

Physically modified starches, such as thermally inhibited starches described in International Publication WO 95/04082, may also be suitable for use herein. Physically modified starches are also intended to include fractionated starches in which there is a higher proportion of amylose.

Chemically modified starches include, without limitation, crosslinked starches, acetylated and organically esterified starches, hydroxyethylated and hydroxypropylated starches, phosphorylated and inorganically esterified starches, cationic, anionic, nonionic, and zwitterionic starches, carboxymethyl starch, and succinate and substituted succinate derivatives of starch. Such modifications are known in the art, for example in MODIFIED STARCHES: PROPERTIES AND USES, Ed. Wurzburg, CRC Press, Inc., Florida (1986).

When crosslinked, the starch is reacted with any crosslinking agent capable of forming linkages between the starch molecules. Typically crosslinking agents suitable herein are those approved for use in foods, such as epichlorohydrin, linear dicarboxylic acid anhydrides, acrolein, phosphorus oxychloride, and soluble metaphosphates. Preferred crosslinking agents are

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phosphorus oxychloride, epichlorohydrin, sodium trimetaphosphate (STMP), and adipic-acetic anhydride, and most preferably phosphorus oxychloride.

The crosslinking reaction itself is carried out according to standard procedures described in the literature for preparing crosslinked, granular starches. Examples of such art include U.S. Patent Nos. 2,328,537 and 2,801,242. Of course, the exact reaction conditions employed will vary with the type of crosslinking agent used, as well as the type of starch base, the reaction scale, etc. The reaction between the starch and the crosslinking agent can be carried out in aqueous medium. In this preferred method, the starch is slurried in water and adjusted to the proper pH, followed by addition of the crosslinking agent.

The amount of crosslinking agent necessary to give a product having the characteristics defined herein will vary depending on, for example, the water fluidity level of the starch, the type of pregelatinization employed, the type of crosslinking agent used, the concentration of the crosslinking agent, the reaction conditions, and the necessity for having a crosslinked starch that falls within a specified range of crosslinking as determined by its viscosity characteristics. One skilled in the art will recognize that it is not the amount of crosslinking agent added to the reaction vessel that determines the properties of the final product, but rather the amount of reagent that actually reacts with the starch, as measure by the Brabender viscosities. Still, the amount of crosslinking agent used for reaction will generally vary from about 0.01% to about 0.07% by weight, depending on the water fluidity of the starch. The exact range can also depend on the pregelatinization process. The type of crosslinking agent used can result in a larger or smaller amount employed. However, in all cases the amount of crosslinking agent must be at least 0.005% by weight.

Any starch or starch blends having suitable properties for use herein may be purified, either before or after any modification or conversion, by any method known in the art to remove starch off flavors, odors, or colors that are native to the starch or created during processing. Suitable purification processes for treating starches are disclosed in the family of patents represented by European Patent No. 554 818. Alkali washing techniques are also useful and described in the family of patents represented by U.S. Patent Nos. 4,477,480 and 5,187,272.

The starch may be used in any amount necessary to achieve the characteristics desired for the particular end use application. In general, the starch is used in an amount of at least about 2% to about 4% by weight of the product.

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Starches suitable for the present invention include amylopectin starches having long outer branches, e.g., waxy corn, where the amylopectin is derived from starch base. Amylose-containing starches from a variety of bases are useful. These amylose-containing starches can be in their native form or lightly converted. Other useful starches include the above amylopectin starches that have been enzyme or acid converted to a lower molecular weight. Also useful are amylose-containing starches that have been crosslinked or dextrinized. A light stabilization ($DS < 0.02$) can be tolerated as long as the starch gels under freezer conditions in the ice cream system. Preferably, the starch is an amylopectin or amylose-containing starch wherein the amylopectin is derived from a starch base having long outer branches. Preferably, the starch base is waxy corn.

Also preferred is a starch that gels during freezing. Gels are defined in many different ways in the art. For the purpose of the present invention, the gel character of a particular ice cream mix after holding in the freezer is quantified based on the measurement of the tangent of the phase angle (" $\tan \delta$ ") for that sample. The $\tan \delta$ for materials varies from values much higher than one to values much lower than one. Those materials which have $\tan \delta > 1$ are typically viewed as liquid-like (under the conditions of measurement) while those with values of $\tan \delta < 1$ are viewed as solid or gel-like. Materials which have $\tan \delta = 1$ are often described as critical gels, meaning that they represent a critical state of matter between liquids and solids. In the present invention, it has been discovered that those ice cream mixes having starches with a $\tan \delta < 1$ after holding in the freezer provide the best resistance to melt-down, and therefore provide the best shape retention of frozen novelties. Accordingly, starches of the present invention that gel during freezing preferably have a $\tan \delta$ less than about one. More preferably, the starches of the present invention result in a $\tan \delta$ of less than about 0.4 when measured in a model ice cream system.

In addition to the starch, the ingredients used for making the frozen dessert include milkfat, milk solids-not-fat, sweeteners, stabilizers, emulsifiers and water. The fat component can be vegetable or animal fat, hydrogenated or otherwise. The vegetable fat can be a mixture of fats, such as palm, coconut or palm kernel oil. The milk solids-not-fat can include powdered or concentrated skimmed milk, as well as powdered or concentrated defatted sweet whey.

Sweeteners commonly used include sucrose, glucose, fructose, corn syrup or corn syrup solids having a dextrose equivalent ("DE") varying from about 20 to about 65. The frozen

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dessert can include a combination of sweeteners. Ingredients can also include colorings and/or flavorings. Further, the frozen dessert can optionally include fruit or fruit pieces, nuts or candies.

Stabilizing agents such as those previously discussed can be used in the formulation of the frozen dessert. Likewise, emulsifiers such as those previously discussed can be used. The stabilizer(s) and emulsifier(s) are used in an amount of about 0.0% to about 1.0%. Typically, as the amount of fat in the frozen dessert decreases, the amount of stabilizer increases.

The proportion of the above ingredients, such as the ratio of starch and sweeteners permits increased stability of the products as well as a reduction in the cost of the products. According to the present invention, these savings are made through the replacement of at least a portion of the sweeteners with the starch. By replacing at least a portion of sweeteners with the starch, the sugar content of the product is reduced while still maintaining its smoothness and creaminess. By at least partially replacing the sweeteners with starch, the melt down of the products is slowed and greater heat shock stability provided.

The process for preparing the frozen desserts is as follows. A mixture of the ingredients is prepared, with the mixture blended for about 15 to about 30 minutes so as to avoid foaming. The mixture is then pasteurized according to standard techniques in the art at about 80°C to about 90°C for about 15 to about 45 seconds. The pasteurized mixture is then homogenized at a pressure of about 2 to about 20 MPa. Preferably, the homogenization is done in two steps, first at a pressure of about 15 to about 20 MPa, and then at a pressure of about 2 to about 4 MPa. The mixture is then cooled to a temperature of about 2°C to about 8°C and aged for a period of about 4 to about 24 hours. The steps of pasteurization, homogenization and cooling can be done in batch steps or continuously. After aging, the mixture is then rapidly frozen at about -2°C to about -8°C to a product overrun of about 80% to about 120%. During freezing, gelation of the starch occurs. This frozen mixture is then hardened to a temperature of about -20°C to about -40°C for a period of about 12 to about 48 hours.

In the examples that follow, all parts and percentages are given by weight and all temperatures in degrees Centigrade (°C) unless otherwise indicated. The following examples are presented to further illustrate and explain the present invention and should not be taken as limiting in any regard. All percents used are on a weight/weight basis. The following analytical and testing procedures were used to characterize the starch products herein.

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EXPERIMENTAL - PROCEDURAL

A. Starches Evaluated

Numerous starches were evaluated for effectiveness as to shape retention (*i.e.*, melt down) and iciness (*i.e.*, formation of ice crystals) in ice cream against a low fat (5%) control and a regular or full fat (10%) commercially available ice cream. Sixteen illustrative starch samples are identified in Table I below –

Table I – Description of Starches

ID #	Short Description	Starch Preparation
1	Native waxy corn starch	unmodified waxy corn starch
2	Acid converted waxy corn starch (20wf)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 20 is obtained
3	Acid converted waxy corn starch (40wf)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 40 is obtained
4	Acid converted waxy corn starch (60wf)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 60 is obtained
5	Acid converted sago starch	sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained
6	Native regular corn starch	unmodified regular corn starch
7	Acid converted sago starch treated with octenyl succinic anhydride	sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained and further treated with OSA to obtain approx 1.75% bound OSA
8	Acid converted waxy corn starch (80wf)	waxy corn starch treated with up to approx 3.0% HCl at 52 C until a desired WF up to 80 is obtained
9	Native waxy rice starch from Bangkok Starch Company	unmodified waxy rice starch
10	Waxy corn starch treated with propylene oxide and phosphorous oxychloride	waxy corn starch treated with PO to obtain approx 6.6% bound PO and further treated with approx 0.004% POC13
11	Tapioca starch treated with propylene oxide and phosphorous oxychloride	Tapioca starch treated with PO to obtain approx 5.0% bound PO and further treated with approx 0.011% POC13
12	Acid converted waxy corn starch treated with propylene oxide	Waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 60 is obtained and further treated with PO to obtain approx 5.2% bound PO
13	Acid converted sago starch treated with propylene oxide	Sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained and further treated with PO to obtain approx 6.4% bound PO
14	Acid converted sago starch treated with sodium tripolyphosphate	Sago starch treated with approx 0.7% HCl at 52 C until a approx 59.5WF is obtained and further treated with STP to obtain approx 0.43% bound PO4
15	Tapioca canary pyrodextrin	Tapioca starch treated with anhydrous hydrogen chloride gas to a pH of approx 2.8 and further dry roasted to a maximum temperature of 154 C and held at temperature until approx 100% solubility and approx 2.2 ABF viscosity is obtained

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10	Waxy corn starch enzymatically debranched	dispersed waxy corn starch treated with approx 6% Promozyme 400L (from Novo) until approx 85% short-chain-amylose is obtained and recovered by spray-drying
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B. Ice Cream Preparation

Three separate low fat (5%) ice cream formulations were prepared according to the formulation and procedure defined below --

Table II – Formulation for Low Fat (5%) Ice Cream Mix

Ingredients	Control Formula (%)	2% Starch Formula (%)	4% Starch Formula (%)
Heavy Cream	13.10	13.10	13.10
Skim Milk	60.80	60.80	60.80
Skim Milk Powder (NFDM)	4.66	4.66	4.66
Sucrose	10.00	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.50	3.50
Corn Syrup Solids DE 24	5.50	4.50	3.50
Stabilizer/Emulsifier Blend	0.44	0.44	0.44
Starch	-	2.00	4.00
Total	100.00	100.00	100.00

Forty (40) percent of the total sucrose and the emulsifier/stabilizer blend were dispersed in the skim milk and mixed for five (5) minutes. A dry blend of NFDM, the remaining sucrose, corn syrup solids and the starch product to be evaluated (as required) was added and mixed for ten (10) minutes. The heavy cream was then added to the mixture, and the mixture gently blended so as to avoid foaming. With all ingredients blended together, the mixture was then pasteurized, homogenized and cooled to 4°C, respectively. Pasteurization was performed at 82°C for thirty (30) seconds, and homogenization was done in two stages, first at 17 MPa and then at 3.4 MPa. Flavoring was then added to the ice cream. The ice cream mixture was aged overnight at 4°C. This mixture was then frozen in a continuous freezer (Technogel 100, available from Wankesha Cherry-Burrell, Delavan, Wisconsin) to an overrun of 100% by keeping the flow rate constant and adjusting the air supply. The mixture was filled into eight ounce (237 ml) sample cups, cooled to -20°C, and then hardened at -30°C for 24 hours. The resulting ice cream contains approximately 5% butterfat, 10% milk solids non-fat ("MSNF"), and 37% solids, based on a calculation using typical nutritional data.

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C. Heat Shock Stability Test

Temperature fluctuation in the distribution chain was simulated by exposing all ice cream samples to 12 temperature cycles in a freezer (model 34-25 commercially available from ScienTemp Corporation, Adrian, Michigan). The freezer was filled with one layer of 100 samples so that airflow could occur around all samples. Cycling conditions were -18°C for 12 hours followed by -6°C for twelve hours.

D. Melt Down Test

An ice cream sample was placed on a screen in a temperature-controlled cabinet at 19°C. A receptacle was placed on a balance below the screen to collect and weigh the meltdown or drip loss (m_d) over a time period of up to four (4) hours. The initial weight of the ice cream (m_0) was determined and kept within a ten percent (10%) standard deviation. Weight of the drip loss (m_d) was recorded over the four-hour time period. Total melt down (MD, %) was determined at the end of the four-hour period according to the following equation –

$$MD = \frac{m_d}{m_0}$$

E. Sensory Evaluation

Samples were evaluated by an expert panel for 10 different attributes as listed and defined below. During each session, two test samples were rated on a scale of 0-15. A control sample that had gone through temperature cycling was used as a base reference and presented with the samples. Each test sample was evaluated twice to reduce the standard deviation.

Blind testing a control product in five (5) different sessions as a test sample determined reproducibility between the panels. This resulted in a standard deviation of 0.5 for each descriptor, confirming sufficient training of the panel.

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DESCRIPTORS

- Firmness -** Force required to compress the sample with teeth and tongue.
- Coldness -** Tendency to cool surfaces of the mouth.
- Cohesiveness -** Tendency to resist loss of structure during chewing. A cohesive product is elastic and maintains structure during chewing. A non-cohesive product is brittle, easily fragments or is crumbly in texture, and provides a clean bite through.
- Rate of Breakdown -** Rate at which a product softens or fragments upon chewing.
- Ice crystals -** Perception of 'grittiness' caused by the number and size of ice crystals. Ideally, no ice crystals can be detected in the mouth.
- Melt Thickness -** Viscosity of film left on the palate.
- Smoothness -** Tendency of the film on the palate to be smooth, slick and non-sticky.
- Gumminess -** Residual film in the mouth is tacky, sticking the tongue to the surfaces of the mouth.
- Vanilla Flavor -** Authentic not of vanilla and cream.
- Off-Note -** Any inappropriate aroma.

F. Rheological Testing Procedure

Each of the ice cream mixes was poured into molds and held in the freezer at -18°C . One mold of each formulation was removed from the freezer and tested on the rheometer as soon as it equilibrated to 25°C . Another mold was removed from the freezer and placed in a constant temperature bath at -6.7°C where it was held for 7-8 days, before it was removed, equilibrated to 25°C and tested on the rheometer.

A sequence of rheology experiments was performed on each sample as described in Table III below -

Table III - Rheology Tests Run on Each Sample

Test Name	Specific Conditions
Dynamic Strain Sweep	$\omega=1\text{rad/s}$, $\gamma_{\text{min}}=.1\%$, $\gamma_{\text{max}}=100\%$
Dynamic Frequency Sweep	$\gamma < \gamma_{\text{cr}}$, $\omega_{\text{min}}=1\text{rads}^{-1}$, $\omega_{\text{max}}=100\text{rads}^{-1}$
Steady Shear Step Rate	Shear rate= 1s^{-1} , length of experiment=120 s
Steady Shear Rate Sweep	Time per shear rate=30s, shear rate range= $1\text{-}100\text{s}^{-1}$

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In Table II, "min" refers to minimum, "max" refers to maximum, "ω" refers to frequency, "γ" refers to strain, "rad" refers to radians, and "s" refers to seconds. Rheological tests described above are according to standard rheological terms.

Experiments on the ice cream mixes in the molds were all done at 25°C. The data tabulated from these experiments includes: G' at 10 rad/s, $\tan \delta$ at 10 rad/s, γ_{cr} , the peak in steady shear viscosity, η at 1/s, the final η after 120s at 1/s, and η at 100/s.

The melt down test used is described above in Section C. The procedure involves placing a known mass of the frozen ice cream on a wire mesh and then recording the weight loss over time. For the current study, the percent weight loss of the ice cream after 4 hours at 19°C was used.

G. Differential Scanning Calorimetry Procedure for Determining T_g'

Differential scanning calorimetry measurements were performed in a Perkin-Elmer DSC-7 with CCA cooling system (Norwalk, Connecticut). Liquid N_2 was used for sub-ambient temperature experiment. The instrument was calibrated with indium and water. An empty stainless-steel pan was used as a reference.

Approximately 10 mg of starch (dry base) was weighed into large volume stainless steel pan. Extra water was added to the sample to reach the desired moisture content. Each starch was tested at two solid levels (40% and 20%) using quench cooling and annealing method to ensure that the measured T_g' and W_g' values are close to real values. The starch/water mixture was first heated from 10°C to 160°C at 10°C/min to ensure that all the starch was fully dispersed in water. Then the starch/water mixture was quench cooled to -70°C and was held for 15 minutes to ensure maximum amount of ice formed. The sample was then heated at 2.5°C/min. to detect the T_g' and the amount of the frozen water. To maximize the amount of ice formed in the ice cream mix, the samples were also subjected to an annealing procedure before temperature scanning. The sample was held at the temperature slightly above T_g' for 30 minutes and then was cooled to below T_g' at 1.0°C/min and held at -40°C for another 10 minutes. The samples were then heated at 2.5°C/min to 20°C. The middle point of the glass transition was reported as T_g' , and the ice melting enthalpy was used to calculate the W_g' value of the starch. Duplicate tests were run for each solid level or cooling condition. The average values from those tests that generated maximum amount of ice were reported.

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EXPERIMENTAL - RESULTS

1. Melt Down and Sensory Results

The effect of the starch according to the present invention on the melt down on ice cream is shown in Figure 1. From Figure 1 it is seen that unmodified or lightly crosslinked starches are preferred. From Figure 1 it is also seen that waxy starches are preferred as the base starch, more preferably, waxy corn starches.

Table IV - Meltdown/Ice Crystal Data on Ice Creams with Starch

Sample ID	Meltdown	Ice Crystals
Starch 16 at 2%	43.00	5.95
Starch 16 at 4%	23.93	4.45
Starch 2 at 2%	16.00	4.7
Starch 3 at 2%	21.00	4.85
Starch 4 at 2%	37.00	4.75
Starch 4 at 4%	4	2.85
Starch 8 at 2%	44.00	4.8
Control ice cream without starch	50	7.5
Starch 12 at 2%	79.00	6.45
Starch 12 at 4%	87.00	3.45
Starch 14 at 2%	25.00	4.8
Starch 13 at 2%	86.00	5.15
Starch 7 at 2%	48.00	4.85
Starch 1 at 2%	14.37	4.7
Starch 6 at 2%	18.73	5.45
Starch 9 at 2%	88.18	5.35
Starch 10 at 2%	80.09	5.1
Starch 5 at 2%	37.10	6.7

The correlation between the reduced melt down of ice cream and the ability of the starch to gel in the model ice cream system under freezer conditions is shown in Figure 2. As can be seen from Figure 2, a starch having a $\tan \delta$ of less than about 1.0 results in a frozen dessert having a melt down of about fifty percent (50%) or less. Starches having a $\tan \delta$ of less than about 0.4 provided the best performance, resulting in an ice cream with the slowest melt down.

As previously noted, the results of the effectiveness of the above samples with respect to melt down are illustrated in Figure 1. Those frozen desserts containing about two to about four percent of a starch that gels during freezing, particularly those starches having a $\tan \delta$ less than about one, resulted in reduced melt down and improved shape retention after exposure to heat shock.

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Those skilled in the art recognize that certain processing conditions (e.g., reduced overrun) and variations in the formula (e.g., higher fat content) can result in improved melt down. The present invention shows that the addition of certain starches improves meltdown, even more than presently used emulsions and stabilizers.

2. Rheological Testing Results

The ice cream mixes show significant changes in rheology during storage at -6.7°C. For most of the samples, tan δ decreases over time, which indicates the development of gelled structures under frozen conditions. Trends are similar for low-fat and non-fat formulas. After 7-10 days, the least gelled (highest tan δ) samples are Sample 16 and Sample 12 while the most gelled (lowest tan δ) samples are Sample 1 and Sample 3. The viscosity of each mix increased or stayed constant throughout the time that the samples were held at -6.7°C. The modulus, G', showed similar behavior to the viscosity.

Table V - Rheology Data on Ice Cream Mixes with Starch

Sample	tan δ at 10 rad/s fresh	η at 1/s fresh	G' at 10 rad/s fresh	tan δ at 10 rad/s @ 7-8 days	η at 1/s 7-8 days	G' at 10 rad/s 7-8 days
Starch 16 at 2%	1.5724	0.2476	0.7279	0.8978	0.9250	3.2386
Starch 16 at 4%	1.2644	0.4205	1.4463	0.472	3.8639	22.018
Starch 2 at 2%	1.6801	0.3120	1.6682	0.2538	15.2720	193.6
Starch 3 at 2%	2.0387	0.2614	0.9887	0.3665	7.9273	52.157
Starch 4 at 2%	2.2618	0.2089	0.6088	0.3597	6.4949	48.769
Starch 4 at 4%	1.8449	0.3314	1.5129	0.3263	46.5130	865.84
Starch 8 at 2%	1.9657	0.2037	0.5129	0.7069	2.8037	12.249
Control ice cream mix without starch	1.7367	0.1982	0.5058	1.6172	0.3039	0.9667
Starch 12 at 2%	2.2580	0.2277	0.686	1.93705	0.2699	0.9439
Starch 12 at 4%	2.0553	0.3717	1.4746	1.6779	0.5326	2.5249
Starch 14 at 2%	0.9583	1.2793	3.9336	0.36985	12.4740	91.049
Starch 13 at 2%	2.1291	0.2271	0.6886	1.3858	0.5244	1.9084
Starch 7 at 2%	1.3449	0.5992	1.7108	0.46385	4.7607	20.386
Starch 1 at 2%	1.9E+00	5.1E-01	2.0E+00	0.31	1.2E+01	8.8E+01
Starch 6 at 2%	7.2E-01	2.6E+00	9.5E+00	0.31	1.8E+01	1.3E+02
Starch 9 at 2%	2.1E+00	4.4E-01	1.8E+00	1.4	7.6E-01	3.1E+00
Starch 10 at 2%	2.1E+00	7.0E-01	2.5E+00	1.5	9.2E-01	4.2E+00
Starch 5 at 2%	1.5E+00	5.2E-01	1.5E+00	0.6	1.8E+00	5.8E+00

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3. Tg' Results

Those starches identified as providing good ice crystal growth inhibition capability included native starches, acid or enzyme converted starches, or chemically modified versions of those starches having a DE less than about 8, and a Tg' less than about -6°C and 'water binding property of Wg' greater than about 0.30 (unfreezable water weight fraction in the unfrozen glass state). Tg' and Wg' are determined by the calorimetry method described above. The results are provided in Table VI below –

Table VI

Starch Sample	Tg' (°C)	Wg'(g/g total glass)
Starch 1	-4.521	0.309
Starch 4	-5.627	0.352
Starch 8	-5.321	0.325
Starch 11	-5.993	0.308
Starch 7	-5.127	0.310
Starch 14	-5.550	0.318
Starch 12	-7.766	0.322
Starch 13	-8.370	0.257
Starch 16	-7.021	0.300
Starch 15	-8.651	0.269

In the present invention, corn syrup solids were reduced to keep the total solids content of the formulation constant. Adding starch to replace fat can affect the glass transition temperature, freezing temperature and the 'water binding' property of the serum phase in the frozen state. Starches were compared at 2% and 4% usage levels. At about 3% to about 4% usage level, the starches of the present invention function in the absence of other stabilizers, providing improved heat shock stability characterized by a reduction in ice crystals as compared to a non-starch control. Higher levels of starch generally further improve performance with respect to melt down and heat shock stability. However, above about 4%, negative textural attributes occur.

4. Low Fat Ice Cream with Starch

Ice cream formulations were prepared according to the formulation defined in Table VI below –

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Table VI - Ice cream formulation 5% butterfat, 3% starch #4, no other stabilizer:

Ingredients	Control Formula (%)	3% Starch Formula (%)
Heavy Cream	13.10	13.10
Skim Milk	60.60	60.60
Skim Milk Powder (NFDm)	4.68	4.68
Sucrose	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.15
Corn Syrup Solids DE 24	5.50	4.15
Emulsifier*	0.44	0.14
Starch 4	-	3.00
Total	100.00	100.00

As in the 'Ice Cream Preparation' section above, when starches of the present invention were added, the 42DE corn syrup solids and the 24DE corn syrup solids were reduced equally to keep the total solids constant. Each ice cream mix was prepared according to the procedure described in the 'Ice Cream Preparation' section.

H. Non-fat Ice Cream Mix with Starch and Stabilizer

Ice cream formulations were prepared according to the formulation defined in Table VII below -

Table VII - Rheological Ice Cream Formulation

Ingredients	1
	0% Fat
Heavy Cream	0.00
Skim Milk	72.67
Non-Fat Dry Milk	4.82
Sucrose	10.50
42DE Corn Syrup Solids	5.78
24DE Corn Syrup Solids	5.78
CREST Stabilizer/Emulsifier	0.46

As in the 'Ice Cream Preparation' section above, when starches of the present invention were added, the 42DE corn syrup solids and the 24DE corn syrup solids were reduced equally to keep the total solids constant. Each ice cream mix was prepared according to the procedure described in the 'Ice Cream Preparation' section. Seven starches were evaluated in the non-fat formula at 2.1% wt/wt.

The ice cream mixes show significant changes in rheology during storage at -6.7°C. For most of the samples, tan δ decreases over time, which indicates the development of gelled structures under frozen conditions. After 7-10 days, the least gelled (highest tan δ) samples are

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Sample 16 and Sample 12 while the most gelled (lowest $\tan \delta$) samples are Sample 1 and Sample 3. The viscosity of each mix increased or stayed constant throughout the time that the samples were held at -6.7°C . The modulus, G' , showed similar behavior to the viscosity.

Table IX – Rheology Data on Non-fat Ice Cream Mixes

Sample	$\tan \delta$ at 10 rad/s fresh	η at 1/s fresh	G' at 10 rad/s fresh	$\tan \delta$ at 10 rad/s @ 7-8 days	η at 1/s 7-8 days	G' at 10 rad/s 7-8 days
Starch 1 at 2.1%	3.9E+00	2.1E-01	4.4E-01	2.6E-01	1.5E+01	1.0E+02
Starch 6 at 2.1%	9.0E-01	1.3E+00	4.2E+00	3.1E-01	8.0E+00	7.4E+01
Starch 10 at 2.1%	3.1E+00	3.2E-01	7.8E-01	2.1E+00	5.0E-01	1.5E+00
Starch 4 at 2.1%	4.0E+00	5.8E-02	1.2E-01	3.8E-01	7.8E+00	4.6E+01
Starch 5 at 2.1%	1.5E+00	4.1E-01	8.4E-01	3.9E-01	2.6E+00	1.4E+01

Although the present invention has been described and illustrated in detail, it is to be understood that the same is by way of illustration and example only, and is not to be taken as a limitation. The spirit and scope of the present invention are to be limited only by the terms of any claims presented hereafter.

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What is claimed and desired to be secured by Letters Patent is:

1. A starch for use in frozen desserts, the starch comprising a $\tan \delta$ of about 1.0 or less, wherein the starch is able to gel during freezing conditions.
2. The starch of claim 1, wherein the starch is used as a sweetener substitute.
3. The starch of claim 1 further comprising a $\tan \delta$ of about 0.4 or less.
4. The starch of claim 1 wherein the starch has been converted to a water fluidity ("WF") of from about 0 to about 80.
5. The starch of claim 4 wherein the starch has been further converted to a WF of from about 20 to about 60. .
6. The starch of claim 1 wherein the starch is an amylopectin starch.
7. The starch of claim 6 wherein the amylopectin starch is waxy corn.
8. The starch of claim 1 wherein the starch provides a reduction in the melt down of the frozen dessert of at least 100% versus frozen desserts without the starch.
9. The starch of claim 1 wherein the starch has a glass transition temperature T_g' of about -6°C or less, a water binding property Wg' of about 0.30 g/g or greater, and is thereby able to provide improved ice crystal inhibition in a frozen dessert versus a frozen dessert without the starch.

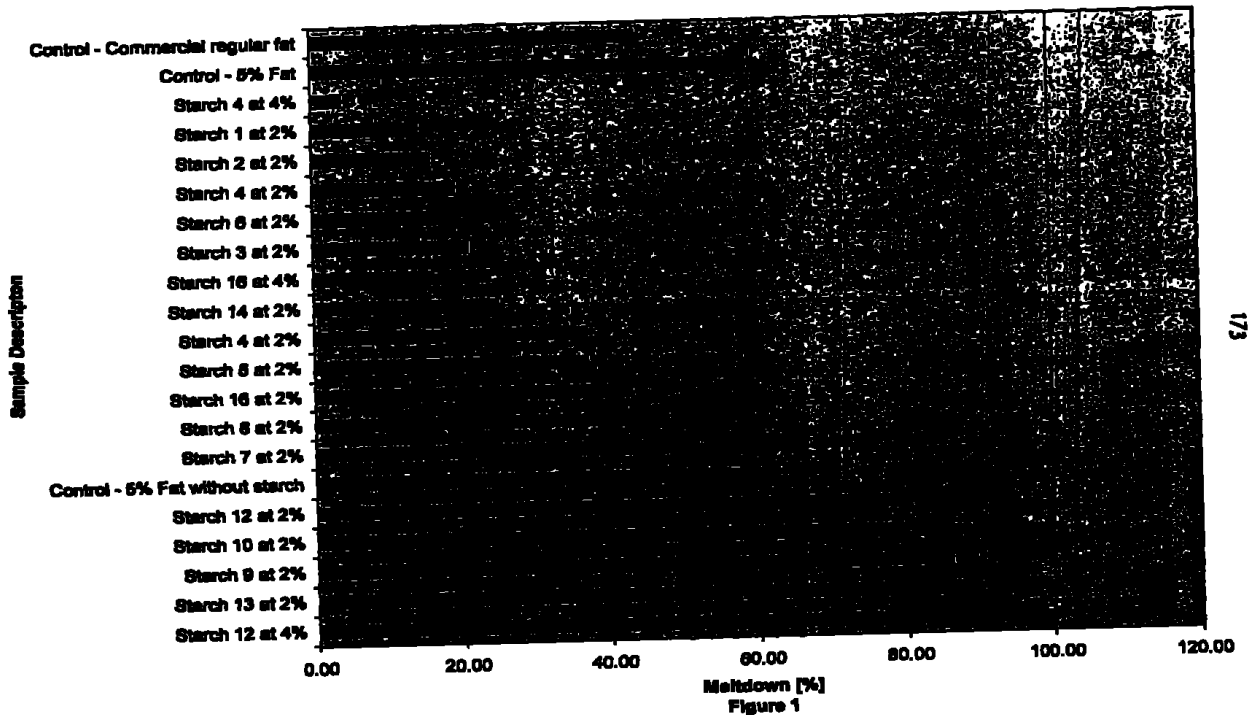
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10. A process for preparing a frozen confectionery, the process comprising the steps of:
- preparing a mixture of ingredients for the frozen confectionery, the mixture including a starch having a $\tan \delta$ of about 1.0 or less,
 - pasteurizing the mixture within a temperature range of from about 80°C to about 90°C;
 - homogenizing the mixture within a pressure range of from about 2MPa to about 20 MPa;
 - cooling the mixture within a temperature range of from about 2°C to about 8°C;
 - aging the mixture for a time period of about 4 hours to about 24 hours;
 - freezing the mixture within a temperature range of from about -2°C to about -8°C; and
 - hardening the mixture at a temperature of from about -20°C to about -40°C;
- wherein the step of freezing effects gelatinization of the starch, and
- wherein the frozen confectionery has an improvement in melt down of at least 100% over frozen confectionery that do not have the starch as an ingredient.
11. The process according to claim 10 wherein the starch is an amylopectin starch.
12. The process according to claim 11 wherein the amylopectin starch is a waxy corn.
13. The process according to claim 10 further comprising the step of replacing at least part of sweetener ingredients of the frozen confectionery with the starch.
14. A frozen dessert comprising a starch having a $\tan \delta$ of about 1.0 or less, wherein the starch gels during freezing conditions.

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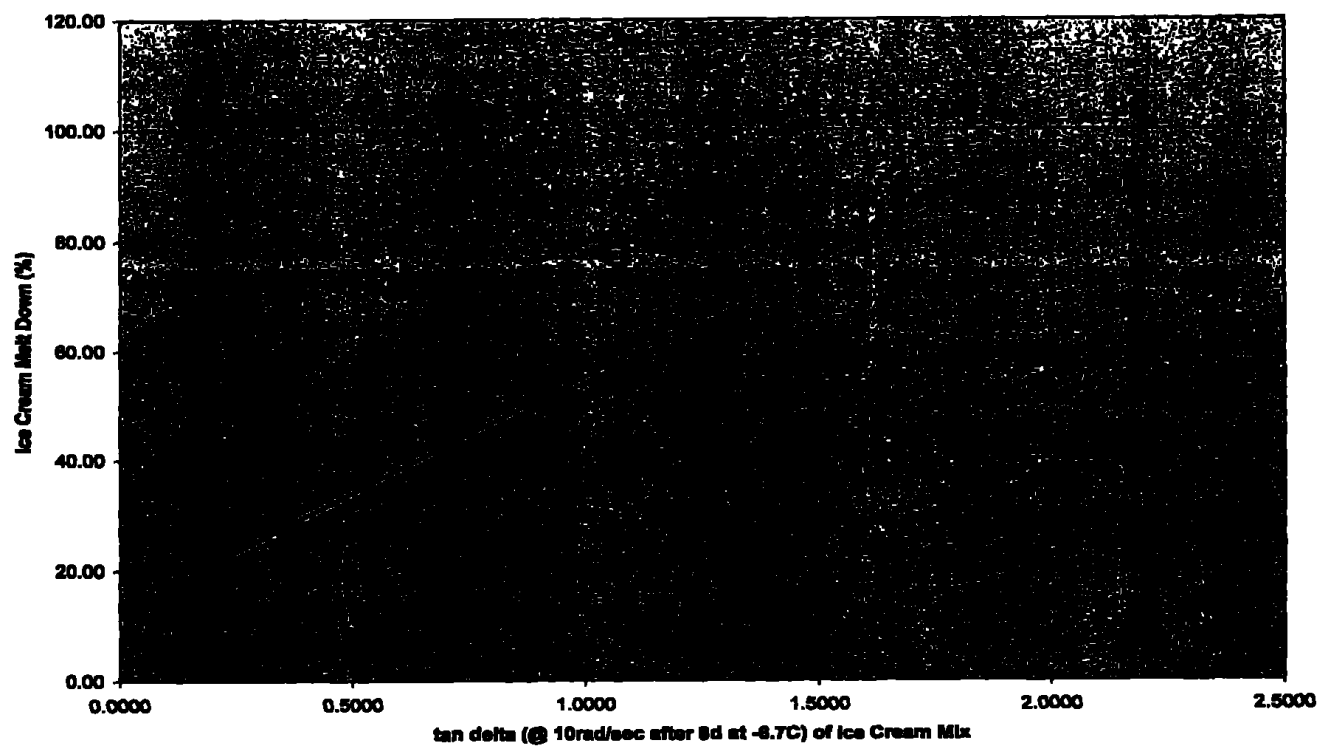
ABSTRACT

Use of starch and starch derivatives in frozen desserts for improved melt down and shape retention. These improved characteristics are retained in the frozen dessert even after multiple heat shock cycling.



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Figure 2



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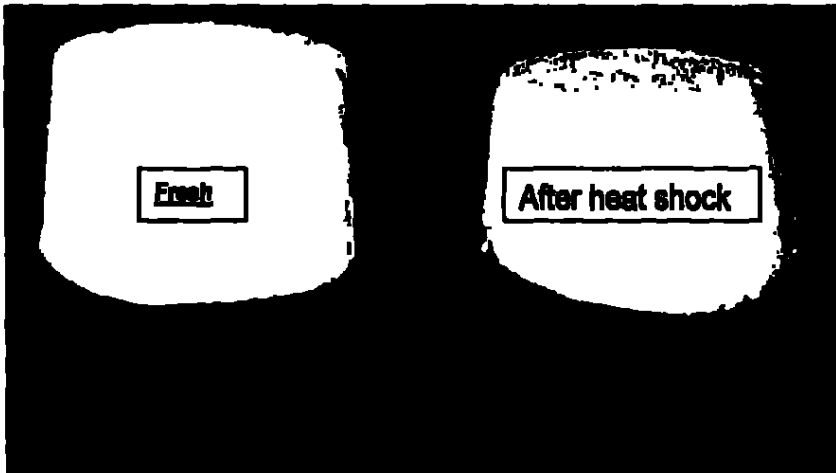


Figure 3. Photo of fresh and heat shocked ice cream containing 3% starch

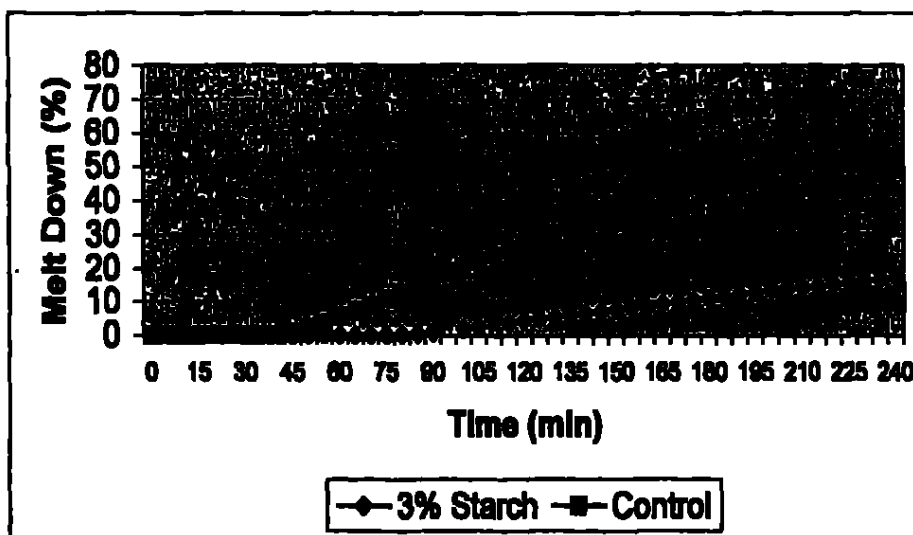


Figure 2. Melt curve of ice cream containing 3% starch

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Lo que se reivindica y para lo cual se desea obtener protección mediante el Título de Patente es lo siguiente:

1. Un almidón para uso en postres congelados, el almidón que comprende una δ "tan" de 1.00 o menor, donde el almidón es capaz de convertirse en gel durante las condiciones de congelamiento.
2. El almidón de la reivindicación 1, en el cual el almidón se utiliza como endulzante sustituto.
3. El almidón de la reivindicación 1, que además comprende una δ "tan" de 0.4 o menor.
4. El almidón de la reivindicación 1, donde el almidón se ha convertido a una fluidez de agua ("FA") de cerca de 0 a cerca de 80.
5. El almidón de la reivindicación 1, donde el almidón se ha convertido posteriormente a una FA de cerca de 20 a cerca de 60.
6. El almidón de la reivindicación 1, donde el almidón es un almidón amilopectina.
7. El almidón de la reivindicación 1, donde el almidón amilopectina es un maíz ceroso.
8. El almidón de la reivindicación 1, donde el almidón suministra una reducción del derretimiento del postre congelado de por lo menos 100% comparado a postres congelados sin el almidón.
9. El almidón de la reivindicación 1, donde el almidón tiene una temperatura de transición vítrea (T_g ') de cerca de -6° C o menor, o una propiedad ligante

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de agua (Wg ') de cerca de 0.30 g/g o mayor, y por ende es capaz de suministrar inhibición mejorada de cristales de hielo en un postre congelado comparado con un postre congelado preparado sin el almidón.

10. Un proceso para preparar confitería congelada, dicho proceso comprendiendo los siguientes pasos:

preparación de una mezcla de ingredientes para la confitería congelada, la mezcla incluyendo un almidón con una δ "tan" de 1.00 o menor,

pasteurizando la mezcla dentro de un rango de temperatura de cerca de 80°C a cerca de 90°C; homogenizando la mezcla dentro de un rango de presión de cerca 2Mpa a cerca de 20Mpa;

enfriando la mezcla en un rango de temperatura de cerca de 2°C a cerca de 8°C;

añejando la mezcla durante un periodo de tiempo de cerca de 4 horas a cerca de 24 horas;

congelando la mezcla dentro de un rango de temperatura de cerca de -2°C a cerca de -8°C, y endureciendo la mezcla a una temperatura de cerca de -20°C a cerca de -40°C;

donde el paso de los efectos de congelamiento y conversión en gel del almidón y donde la confitería congelada muestra un derretimiento mejorado de por lo menos 100% comparada con confitería congelada que no tiene el almidón como ingrediente.

11. El proceso, de acuerdo con la reivindicación 10 donde el almidón es un

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almidón amilopectina.

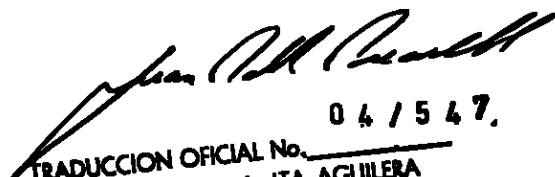
12. El proceso, de acuerdo con la reivindicación 11, donde el almidón amilopectina es un maíz ceroso.

13. El proceso, de acuerdo con la reivindicación 10 que además comprende el paso de reemplazar por lo menos parte de los ingredientes endulzantes de la confitería congelada con el almidón.

14. Un postre congelado que comprende un algodón con una δ "tan" de 1.00 o menor, donde el almidón es capaz de convertirse en gel durante las condiciones de congelamiento.

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JUAN CARLOS ARIZA AGUILERA
TRADUCTOR E INTERPRETE OFICIAL
Reg. 118 DE MAYO 2003
DEL MIN. INTERIOR Y DE JUSTICIA

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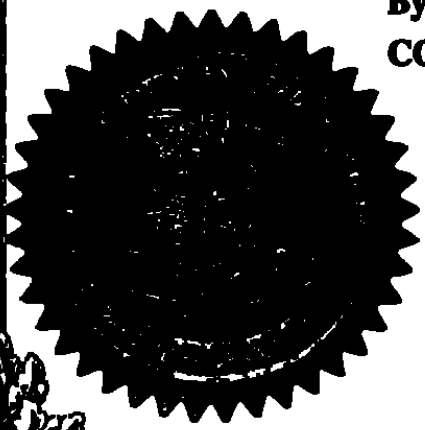
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APPLICATION NUMBER: 10/926,555

FILING DATE: August 26, 2004

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**UTILITY
PATENT APPLICATION
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(Only for new nonprovisional applications under 37 CFR 1.53(a))

Attorney Docket No.:	3048.FDI
First Inventor:	Kosholt, et al.
Title:	STARCH FOR FROZEN DESSERTS
Express Mail Label No.	EL 869398714 US

22386 U.S. PTO
10/926555



APPLICATION ELEMENTS

ADDRESS TO: Mail Stop Patent Application
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

1. ☒ Fee Transmittal Form (e.g., PTO/SB/17)
(Submit an original, and a duplicate for fee processing)
2. ☐ Applicant claims small entity status.
See 37 CFR 1.27.
3. ☒ Specification [30 Total Pages]
(preferred arrangement set forth below)
 - Descriptive title of the invention
 - Cross Reference to Related Applications
 - Statement Regarding Fed sponsored R & D
 - Reference to sequence listing, a table,
Or computer program listing appendix
 - Background of the invention
 - Brief Summary of the invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
 - Abstract of the Disclosure
4. ☒ Drawing(s) (35 USC 113) [Total Sheets 8]
5. ☒ Oath or Declaration [Total Pages 2]
 - a. ☒ Newly executed (original or copy) - Unsworn
 - b. ☐ Copy from a prior application (37 CFR 1.63(d))
(for continuation/divisional with Box 18 completed)
 - c. ☐ DELETION OF INVENTOR(S)
Signed statement attached deleting
inventor(s) named in the prior application,
see 37 CFR 1.63(d)(2) and 1.33(b).

7. ☐ CD-ROM or CD-R in duplicate, large table or
Computer Program (Appendix)
8. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
 - a. ☐ Computer Readable Form (CRF)
 - b. Specification Sequence Listing on:
 - i. ☐ CD-ROM or CD-R (2 copies); or
 - ii. ☐ paper
 - c. ☐ Statements verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

9. ☐ Assignment Papers (cover sheet & document(s))
10. ☐ 37 CFR 3.73(b) Statement ☐ Power of Attorney
(when there is an assignee)
11. ☐ English Translation Document (if applicable)
12. ☐ Information Disclosure ☐ Copies of IDS
Statement (IDS)/PTO-1449 Citations -
13. ☐ Preliminary Amendment
14. ☒ Return Receipt Postcard (NPEP 603)
(Should be specifically itemized)
15. ☐ Certified Copy of Priority Document(s)
(If foreign priority is claimed)
16. ☐ Request and Certification under 38 U.S.C. 122
(b)(2)(B)(i). Applicant must attach form PTO/SB/38
or its equivalent.
17. ☐ Other

6. Application Data Sheet. See 37 CFR 1.78

18. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a Preliminary Amendment, or in an Application Data Sheet 37 CFR 1.78:

☐ Continuation ☐ Divisional ☐ Continuation-in-Part (CIP) of prior application No. ____/_____
Prior application information: Examiner _____ Group/Art Unit: _____

For CONTINUATION or DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or Declaration is supplied under Box 5b, is considered a part of the disclosure of the accompanying Continuation or Divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

19. CORRESPONDENCE ADDRESS

☒ Customer Number or Bar Code Label

or ☐ Correspondence address below

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Signature				Date	8/28/04

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STARCH FOR FROZEN DESSERTS

5 CROSS REFERENCE TO RELATED APPLICATIONS

The present application claims the benefit of U.S. Provisional Application Nos. 60/498,834 and 60/511,765, filed 29 August 2003 and 16 October 2003, respectively.

BACKGROUND OF THE INVENTION

10 **Technical Field.** The present invention relates to a starch for use in frozen food products. More specifically, the present invention is directed towards a functional starch or starch derivative for use in frozen desserts in order to obtain improved structure characteristics, including slow melt down, excellent shape retention, reduced iciness, greater heat shock stability and reduced expansion and contraction of the desserts due to variations in altitude or pressure.

15 **Background Information.** In addition to their rich flavor, frozen confectioneries are enjoyed for their creaminess and smoothness. However, in order to preserve these characteristics, these products have to be handled and stored with care. Unfortunately, even small temperature variations can occur during storage, distribution and handling. For example, such variations can occur when a consumer buys a frozen product and does not consume it right away, as in the time from when a consumer purchases such a product at a grocer and places that product in his freezer. Partial or even substantial defrosting of the product can occur before it is refrozen. This temperature cycling can result in ice crystal growth in the product, as well as loss in product shape. Such growth affects both the visual appearance and organoleptic properties of the frozen product, thereby reducing its quality and appeal, at least as perceived by the consumer.

25 The most frequently occurring textural defect in ice cream is due to ice crystal growth, and is the primary limitation to its shelf life. As such, it is desirable for frozen desserts to be stable against 'heat shock', or the cyclic conditions of partial thawing and refreezing that occur during typical storage, shipping and handling of these products.

30 As used herein, a frozen dessert, frozen confectionery or frozen product refers to a product in which water is present in both the liquid and frozen state. Examples of frozen deserts

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include ice cream, ice milk, water ice and parfaits. As used herein, frozen novelties refer to frozen desserts such as those available on a stick or in sandwiches or cones.

Ice crystals in frozen products need to be numerous and of small, nearly uniform size to avoid detection when eaten. Because ice crystals are relatively unstable, during storage they undergo changes in number, size and shape, which is collectively referred to as recrystallization. While some recrystallization occurs naturally at constant temperatures, by far the majority of it occurs due to temperature fluctuations or heat shock. An increase in temperature during the frozen storage of a product such as ice cream causes some of the ice crystals, particularly the smaller ones, to melt. As the temperature decreases, that water refreezes without renucleation, instead depositing on the surface of larger crystals. As such, heat shock or temperature cycling in frozen products reduces the total number of ice crystals and increases the mean ice crystal size.

To a consumer, the perception of coldness and iciness of ice cream is directly related to the amount of ice formed in the ice cream and the size of the ice crystals. Typically, consumers can detect ice crystals larger than about fifty (50) microns. Such large ice crystals contribute to the coarseness and iciness of the ice cream. For a typical ice cream, the amount of ice formed and the crystal size distribution are mainly determined by the formulation, processing, and storage conditions as outlined below.

Ice crystallization is also affected by the glass transition temperature (T_g') and the freezing point of the ice cream. In a maximally freeze-concentrated solution, an invariant condition occurs at temperature T_g' and concentration C_g' (or unfreezable water W_g'), depending on the particular mixture of compounds in the formulation. The unfreezable water W_g' is that water rendered unfreezable in the maximum freeze-concentrated solution due to the low diffusivity in the glassy state. When ice cream is stored below T_g' , the system is in the glassy state. In this glassy state, the diffusion-controlled processes that typically result in reduced quality and stability can be largely inhibited. Above T_g' , the system is in a rubbery state where the translational diffusion is free to occur. The rate of diffusion-controlled deterioration increases exponentially with increasing ΔT , the temperature difference between storage temperature T and T_g' ($\Delta T = T - T_g'$), in agreement with William-Landel-Ferry ("WLF") kinetics.

Various stabilizing gums have been used as additives in an attempt to improve the heat shock stability and creaminess of frozen food products. Such stabilizing gums have included

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gelatin, agar, gum acacia, guar gum, carob or guar seed flour, locust bean gum, carrageenan, alginate, carboxymethyl cellulose, xanthan and the like, with each exhibiting their own set of advantages and disadvantages. Microcrystalline cellulose ("MCC") and carboxymethyl cellulose are often used in combination with other stabilizing gums to improve functional effectiveness.

5 MCC and cellulose gum can be used together to reduce ice crystal growth. However, MCC can continue to activate during aging or subsequent processing, resulting in unanticipated viscosity.

While gums improve stability, their use has several disadvantages. For example, the amount of stabilizing gum required to provide heat shock stability can result in a product having an unacceptably greasy and/or gummy mouthfeel. Further, products containing such stabilizing compounds are regulated and often poorly perceived by the public. In addition, these additives can be expensive. Several attempts have been made to replace stabilizers in frozen products by replacing them at least in part with other components. However, stabilizers have the added benefit of reducing the size of ice crystals in those products. By replacing all or part of those stabilizers, the size of ice crystals in the product can increase, resulting in a less desirable
15 product.

Emulsifiers provide stability and creaminess to frozen products by facilitating an interface between the aqueous phase and the fat phase of the product. Milk or milk proteins and eggs yolks are natural sources of emulsifiers useful for their water-binding properties. Commercially available emulsifiers are generally derived by chemical reactions with naturally occurring glycerides.

Emulsifiers added to ice cream reduce the stability of this fat emulsion by replacing proteins on the surface of the milkfat. For example, ice cream is a frozen dessert that is both an emulsion and foam. When an ice cream mixture is whipped, the fat emulsion begins to break down and the fat globules begin to flocculate or destabilize. This partially coalesced fat
25 stabilizes air bubbles that are whipped into this mix. Without emulsifiers, the fat globules would be able to resist this coalescing due to the proteins being absorbed to the fat globule, causing the air bubbles to not be properly stabilized and affecting the texture or smoothness of the ice cream.

Emulsifiers also affect the melt down of the ice cream. When ice cream is placed in an ambient environment, the ice cream melts and the fat-stabilized foam structure collapses.
30 Melting is controlled by outside temperature and rate of heat transfer. However, even after the ice crystals melt, the ice cream does not collapse until the fat-stabilized foam structure collapses.

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This collapse is a function of the extent of fat destabilization/partial coalescence, which is controlled by numerous factors, including but not limited to, emulsifier concentration, the type of emulsifier, processing conditions and so forth.

5 The frozen product ice cream is basically composed of milkfat, serum solids or milk solids-non-fat, sweeteners, stabilizers and emulsifiers, and water. Stabilizers and emulsifiers were previously discussed. Water found in ice cream comes from the milk or other ingredients or is added. The milkfat or butterfat is typically obtained from sweet or heavy cream, and provides flavor, smooth texture and body to the ice cream. Other types of fats, such as palm oil, can also be used. The serum solids contain the lactose, caseins, whey proteins, minerals and ash content, and can be found in concentrated skimmed milk or skim milk powder. Sucrose is typically the main sweetener. At least a portion of the sweetener can be obtained from corn syrup solids, which provides a firmer and chewier body to the ice cream. Liquid and solid corn syrup is available in various dextrose equivalents ('DE'), which is a measure of the reducing sugar content of the syrup calculated as dextrose and expressed as a percentage of the total dry weight. As the DE increases, the sweetness increases and the molecular weight decreases.

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The formulation used for manufacturing frozen desserts varies depending upon the type of product desired. Types of frozen products include economy brands typically having the legal minimum fat and total solids content, standard brands, premium brands and super premium brands, as well as a hard frozen, soft frozen, low fat, light, sherbet, sorbet or frozen yogurt product. As the amount of fat increases in the formulation, the frozen product becomes less viscous.

Ice cream ingredients are blended together according to the formulation selected to produce the ice cream mix. The mix is then pasteurized, followed by homogenization, or vice versa. Once homogenized and pasteurized, the mix is aged to allow the fat to cool and crystallize, and the proteins and polysaccharides to fully hydrate. After aging, the mix is frozen, during which time particulates such as fruit, nuts, candy, cookies, etc. may be added to the mix, followed by hardening of the ice cream, typically at temperatures of -30 to -40°C. The freezing and hardening is best done at a fast rate, as a fast freezing rate promotes the formation of many small ice crystals instead of fewer larger ice crystals.

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30 The temperature at which ice cream freezes is affected or depressed by the concentration of sweeteners and/or their molecular weight. For example, a mix having a higher concentration

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of sugars and/or sugars of a lower molecular weight will have a lower freezing temperature than a mix having a lower concentration of sugars and/or sugars of a higher molecular weight. The lower the freezing temperature, the softer the ice cream, the greater the freeze out of the water during hardening, and the greater the susceptibility of the ice cream to heat shock.

5 For low fat or reduced fat frozen desserts, particularly frozen novelties, shape retention is necessary for product quality. It is known that melt characteristics of frozen desserts are relevant to shape retention. For ice cream, melt down is strongly influenced by the fat and air domains of the ice cream. As explained above, both fat and emulsifiers in the dessert affect melt down. For a low or reduced fat formula, melt down can be critical. Simply adding more emulsifier to make up for the loss of fat can be expensive and affect the characteristics of the ice cream, and is therefore not a preferred solution.

10 Frozen desserts are also affected by altitude. When frozen desserts such as ice cream or frozen novelties are transported from a low altitude to a higher altitude (e.g., from sea level to a mile-high elevation), the reduction in atmospheric pressure causes the air cells in the frozen
15 dessert to expand and collapse. In the case of ice cream, this results in the ice cream pushing out of the container, often popping off the lids (expansion) and pulling away from the sides of the container (shrinkage). For frozen novelties, this can result in cracked or broken chocolate coatings and/or separation from the coating. Understandably, such product defects are unacceptable from a consumer standpoint.

Various starch-based products have been marketed and sold for use in frozen desserts. These include converted starches, stabilized starches and crosslinked starches. It is well known in the food industry to use starch as a thickener and/or binder, i.e., as a stabilizer. Typically, starches have been used in frozen confectioneries as a fat substitute or texturing agent. With the
25 trend toward diminishing sugar in frozen products such as ice cream, starch-based products such as hydrogenated starch hydrolysates (polyols) have proven useful alternatives to sweeteners such as maltodextrin and polydextrose. These starch-based substitutes are typically the liquid and/or solid corn syrups previously discussed. However, these substitutes do not function in reducing melt down and/or reducing iciness in frozen desserts, nor do they function in controlling expansion or contraction of those desserts.

30 Accordingly, there is a need for a starch product for use in frozen desserts that functions in reducing the melt down of the dessert when exposed to temperature fluctuations during

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storage and distribution, thereby aiding in retaining the shape of the frozen product, particularly novelty products. Further, there is a need for a starch product for use in frozen desserts that functions in reducing the risk of ice crystal growth during temperature cycling. Finally, there is a need for a starch product for use in frozen desserts that functions in reducing the expansion and shrinkage of the product when transported at high altitudes.

SUMMARY OF THE INVENTION

The present invention is directed towards functional starch and/or starch derivatives useful in frozen desserts for obtaining improved structure characteristics, including a reduction in melt down, improved shape retention and/or decrease in ice crystal growth during temperature cycling. The invention is also directed towards a frozen dessert that includes those functional starch and/or starch derivatives. The frozen dessert can include fat, sweeteners, milk solids-not-fat, stabilizers, emulsifiers, water and starch. The functional starch and/or starch derivative of the present invention can be used to replace at least a portion of any of the other solids, or can be added on top of those solids.

In one aspect, the invention includes a frozen dessert formulated with at least one functional starch, wherein the at least one starch is able to gel during freezing conditions and the frozen dessert mix or formulation has a $\tan \delta$ of about 1.0 or less. In another aspect, the frozen dessert mix or formulation has a $\tan \delta$ of about 0.4 or less. In one aspect of the present invention, the at least one starch is a degraded or converted starch having a water fluidity ("WF") of from about 0 to about 80. In another aspect, the starch has been converted to a WF of from about 20 to about 65. In one aspect, at least one starch used in the formulation is an amylopectin starch. Natural (base) starches typically are a mixture of amylose and amylopectin. Amylopectin starches typically refer to those starches containing at least about 60 % by weight amylopectin. This amylopectin starch can be a waxy starch (i.e., no more than about 10% amylose by weight), including waxy corn (waxy maize). The functional starch, when added to the frozen dessert, provides an improved reduction in the melt down of the frozen dessert versus a frozen dessert formulated without the functional starch. For example, a frozen dessert prepared with the functional starch can have a melt down of 80% or less versus frozen desserts that is completely melted down (100%) when formulated without the starch over the same time frame.

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In one aspect, the invention includes a frozen dessert formulated with at least one functional starch, wherein the starch gels quickly upon freezing the dessert mix containing the starch. In another aspect the quick gelling functional starch is an amylose-containing starch. In a further aspect the invention includes a frozen dessert formulated with at least one functional starch, wherein the starch gels in the dessert mix after repeated exposure to temperature cycling between, e.g., about -18°C and about -6.7°C. In another aspect the functional starch that gels when exposed to such temperature cycling is a waxy starch. The gelling behavior of such useful functional starches can be determined by measuring the $\tan \delta$ of the dessert mix prepared with the starch after holding the mix, e.g., at about -6.7°C for approximately seven to ten days.

The frozen dessert includes ice cream, ice milk, water ice and parfaits. In one aspect, the frozen dessert is ice cream. In another aspect, the frozen dessert is low fat ice cream. The frozen dessert also includes frozen novelties that can be manufactured and sold in various shapes. The starch functions in providing shape retention for the novelty. In another aspect, the starch functions in providing shape retention when the novelty is heat shock treated, e.g., for up to 12 freeze/thaw cycles, with each cycle occurring over a twenty-four hour period.

In one aspect, useful starches for frozen desserts have a glass transition temperature (T_g) of about -6°C or greater. In another aspect, useful starches have a water binding property (W_g) of about 0.30 g/g or greater. The starches of the present invention are able to provide improved ice crystal inhibition in a frozen dessert as compared to a frozen dessert without the starch.

A process for preparing frozen confectioneries is also provided. The process includes the steps of preparing a mixture of ingredients for the frozen confectionery wherein the mixture has a $\tan \delta$ of about 1.0 or less when it includes at least one functional starch, and freezing the mixture within a temperature range of from about -2°C to about -8°C wherein the freezing effects gelling of the starch. Frozen confectioneries prepared according to this process have an improvement in meltdown over similar frozen confectioneries that do not have the functional starch as an ingredient. For example, frozen confectioneries prepared with the functional starch can have an improvement (reduction) in meltdown of at least 80%. The process can also include the step of pasteurizing the mixture within a temperature range of from about 70°C to about 100°C. In a further aspect, the process includes the step of homogenizing the mixture within a pressure range of from about 2 MPa to about 20 MPa. In an additional aspect, the process includes the step of cooling the mixture within a temperature range of from about 2°C to about

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8°C. The process can also include the step of aging the mixture for a time period of about 4 hours to about 24 hours. The process can include the step of hardening the mixture at a temperature of from about -20°C to about -40°C. The process can also include the step of replacing at least part of solids ingredients of the frozen confectionery with the functional starch.

5 The present invention further provides for a functional starch useful in frozen desserts. The frozen dessert includes a $\tan \delta$ of about 1.0 or less when formulated with the functional starch. In another aspect, the functional starch is able to gel during freezing conditions.

10 The present invention further provides for a functional starch for use in frozen desserts wherein the starch provides the frozen dessert a reduced amount of expansion and shrinkage of the ice cream when exposed to a defined vacuum as compared to a frozen dessert without the starch. Such a functional starch is beneficial commercially where the frozen desserts are transported over high altitudes.

BRIEF DESCRIPTION OF THE DRAWINGS

15 The manner in which these objectives and other desirable characteristics can be obtained is explained in the following description and attached drawings in which:

Figure 1 is a graph illustrating the percent melt down of various ice creams after four hours at 19°C.

20 Figure 2 is a graph illustrating the correlation between the melt down of ice cream and the $\tan \delta$ of the mix after seven to eight days at -6.7°C.

Figure 3 is a photograph illustrating the effect in melt down of an ice cream containing a starch of the present invention after heat shock treatment as compared to freshly prepared ice cream.

25 Figure 4 is a melt down curve of an ice cream containing 3% of a starch according to the present invention as compared to a control ice cream containing no starch.

Figure 5 is a series of six photographs illustrating the effect of altitude on ice cream containing 3% of a starch according to the present invention as compared to a control ice cream containing no starch.

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DETAILED DESCRIPTION OF THE INVENTION

The starch base material used for the present invention may be derived from any source, including cereal or root starches. Typical sources for the starches are cereals, tubers, roots, legumes and fruits. Native starch sources include, for example, any variety of corn (maize), pea, potato, sweet potato, banana, barley, wheat, rice, oat, sago, amaranth, tapioca, arrowroot, canna, sorghum and waxy and high amylose varieties thereof. As used herein, "waxy" includes starches containing no more than about 10% amylose by weight. As used herein, the term "high amylose" includes starches containing at least about 40% by weight amylose. As used herein, the term "amylose-containing" includes those starches containing at least about 10% by weight amylose. Preferably, the starch base material is an amylopectin starch such as a waxy corn starch or amylose-containing starch.

The granular starch base can be one that has been lightly converted or hydrolyzed to a water fluidity ("WF") of about 0 to about 80. The term "water fluidity" has a very specific meaning as described further herein below. The starch is converted to its fluidity or thin-boiling form using a suitable method of degradation that results in the modified starch defined herein. Such degradation includes, for example, mild acid hydrolysis with an acid such as sulfuric or hydrochloric acid, conversion with hydrogen peroxide, or enzyme conversion. Converted starch products can include blends of different starches converted by various techniques as well as converted starch(es) blended with unconverted starch(es). Commercially, starch is typically converted by acid or enzyme conversion techniques. Hydrogen peroxide can also be used to convert or thin the starch, either alone or with metal catalysts.

As noted above, the starch of the present invention can be converted to water fluidity ("WF") of from about 0 to about 80, particularly from about 20 to about 65. Water fluidity, as used herein, is an empirical test of viscosity measured on a scale of 0-90 wherein fluidity is inversely proportional to viscosity. Water fluidity of starches is typically measured using a rotational shear-type viscometer (commercially available from Thomas Scientific, Swedesboro, New Jersey), standardized at 30°C with standard oil that has a viscosity of 24.73 cps and requires 23.12±0.05 sec for 100 revolutions. Accurate and reproducible measurements of water fluidity are obtained by determining the time which elapses for 100 revolutions at different solids levels depending on the starch's degree of conversion – as the degree of conversion increases, the viscosity decreases and the WF values increase.

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The base material can be modified either chemically or physically using techniques known in the art. The modification can be to the base or the converted starch, though typically the modification is carried out after conversion.

5 Physically modified starches, such as thermally inhibited starches described in International Publication WO 95/04082, may also be suitable for use herein. Physically modified starches are also intended to include fractionated starches in which there is a higher proportion of amylose.

10 In another embodiment, cold water soluble ('CWS') starches may be suitable for use. CWS starches are pre-gelatinized, cold water swelling, or cold water dispersible starches. CWS starches have been gelatinized and dried by the manufacturer before sale to the customer in a powdered form. They can be made by drum drying, spray drying or extrusion of either native or modified starch. They develop viscosity when dispersed in cold or warm water without the need for further heating. Pre-gelatinized starch is also known as precooked starch, pregelled starch, instant starch, cold water soluble starch, or cold water swelling starch.

15 Chemically modified starches include, without limitation, crosslinked starches, acetylated and organically esterified starches, hydroxyethylated and hydroxypropylated starches, phosphorylated and inorganically esterified starches, cationic, anionic, nonionic, and zwitterionic starches, carboxymethyl starch, and succinate and substituted succinate derivatives of starch. Such modifications are known in the art, for example in MODIFIED STARCHES: PROPERTIES AND
20 USES, Ed. Wurzburg, CRC Press, Inc., Florida (1986).

When crosslinked, the starch is reacted with any crosslinking agent capable of forming linkages between the starch molecules. Typically crosslinking agents suitable herein are those approved for use in foods, such as epichlorohydrin, linear dicarboxylic acid anhydrides, acrolein, phosphorus oxychloride, and soluble metaphosphates. Preferred crosslinking agents are
25 phosphorus oxychloride, epichlorohydrin, sodium trimetaphosphate (STMP), and adipic-acetic anhydride, and most preferably phosphorus oxychloride.

The crosslinking reaction itself is carried out according to standard procedures described in the literature for preparing crosslinked, granular starches. Examples of such art include U.S. Patent Nos. 2,328,537 and 2,801,242. Of course, the exact reaction conditions employed will
30 vary with the type of crosslinking agent used, as well as the type of starch base, the reaction scale, etc. The reaction between the starch and the crosslinking agent can be carried out in

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aqueous medium. In this preferred method, the starch is slurried in water and adjusted to the proper pH, followed by addition of the crosslinking agent.

The amount of crosslinking agent necessary to give a product having the characteristics defined herein will vary depending on, for example, the water fluidity level of the starch, the type of pregelatinization employed, the type of crosslinking agent used, the concentration of the crosslinking agent, the reaction conditions, and the necessity for having a crosslinked starch that falls within a specified range of crosslinking as determined by its viscosity characteristics. One skilled in the art will recognize that it is not the amount of crosslinking agent added to the reaction vessel that determines the properties of the final product, but rather the amount of reagent that actually reacts with the starch, as measure by the Brabender viscosities. Still, the amount of crosslinking agent used for reaction will generally vary from about 0.01% to about 0.07% by weight, depending on the water fluidity of the starch. The exact range can also depend on the pregelatinization process. The type of crosslinking agent used can result in a larger or smaller amount employed. However, in all cases the amount of crosslinking agent should be at least 0.005% by weight.

Any starch or starch blends having suitable properties for use herein may be purified, either before or after any modification or conversion, by any method known in the art to remove starch off flavors, odors, or colors that are native to the starch or created during processing. Suitable purification processes for treating starches are disclosed in the family of patents represented by European Patent No. 554 818. Alkali washing techniques are also useful. Examples of such washing techniques are described in the family of patents represented by U.S. Patent Nos. 4,477,480 and 5,187,272.

The starch may be used in any amount necessary to achieve the characteristics desired for the particular end use application. In general, the starch is used in an amount of at least up to about 4% by weight of the product.

Starches suitable for the present invention include amylopectin starches having long outer branches, e.g., waxy corn, where the amylopectin is derived from starch base. Amylose-containing starches from a variety of bases are also useful. Any of these amylopectin and amylose-containing starches can be in their native form, lightly converted, crosslinked, and/or dextrinized. Other useful starches include the above starches that have been enzyme or acid converted to a lower molecular weight. A light stabilization ($DS < 0.02$) can be tolerated as long

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as the starch gels under freezer conditions in the ice cream system. Preferably, the starch is an amylopectin or amylose-containing starch wherein the amylopectin is derived from a starch base having long outer branches. Preferably, the starch base is waxy corn.

Also preferred is a starch that gels during freezing. Gels are defined in many different ways in the art. For the purpose of the present invention, the gel character of a particular ice cream mix after holding in the freezer is quantified based on the measurement of the tangent of the phase angle ("tan δ ") for that sample. The tan δ for materials varies from values much higher than one to values much lower than one. Those materials which have tan $\delta > 1$ are typically viewed as liquid-like (under the conditions of measurement) while those with values of tan $\delta < 1$ are viewed as solid or gel-like. Materials which have tan $\delta = 1$ are often described as critical gels, meaning that they represent a critical state of matter between liquids and solids. In the present invention, it has been discovered that those ice cream mixes containing starches have a tan $\delta < 1$ after holding in the freezer at -6.7°C provide the best resistance to melt-down, and therefore provide the best shape retention of frozen novelties. Further, such functional starch containing mixes are able to reduce expansion and contraction of the frozen dessert when exposed to variations in atmospheric pressure, such as transportation of the product at high altitudes. Accordingly, starches of the present invention that gel during freezing preferably result in a tan δ less than about one when measured in a model ice cream system. More preferably, the starches of the present invention result in a tan δ of less than about 0.4 when measured in a model ice cream system.

Typically, the Wg' of carbohydrates decreases as the Tg' value increases. Starches that have a high Tg' and low Wg' often cannot provide adequate ice crystal growth inhibition without causing serious negative impact on texture. Accordingly, a series of starch samples from various botanical sources having different chemical substitutions, treatment, and molecular weight were measured for their Tg' and Wg' values in water. In one aspect, inhibition of ice crystal growth in frozen dessert during temperature cycling was provided with starches having higher Tg' and Wg' values. Those starches having a Tg' higher than -6°C and a Wg' higher than 0.30 in water provide significant ice crystal reduction when used at 2-4% in the ice cream formulation described *supra*.

In addition to the starch, the ingredients used for making the frozen dessert include milkfat, milk solids-not-fat, sweeteners, stabilizers, emulsifiers and water. The fat component can be vegetable or animal fat, hydrogenated or otherwise. The vegetable fat can be a mixture of

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fats, such as palm, coconut or palm kernel oil. The milk solids-not-fat can include powdered or concentrated skimmed milk, as well as powdered or concentrated defatted sweet whey.

Sweeteners commonly used include sucrose, glucose, fructose, corn syrup or corn syrup solids having a dextrose equivalent ("DE") varying from about 20 to about 65. The frozen dessert can include a combination of sweeteners. Ingredients can also include colorings and/or flavorings. Further, the frozen dessert can optionally include fruit or fruit pieces, nuts or candies.

Stabilizing agents such as those previously discussed can be used in the formulation of the frozen dessert. Likewise, emulsifiers such as those previously discussed can be used. The stabilizer(s) and emulsifier(s) are used in an amount of about 0.0% to about 1.0%. Typically, as the amount of fat in the frozen dessert decreases, the amount of stabilizer increases.

The proportion of the above ingredients, such as the ratio of starch and sweeteners permits increased stability of the products. According to the present invention, this stability is made through the replacement of at least a portion of the total solids with starch. By replacing at least a portion of the solids with starch, the solid content of the product is maintained while maintaining the smoothness and creaminess of the product. By at least partially replacing the solids with starch, the melt down of the products is slowed and greater heat shock stability provided.

The process for preparing the frozen desserts is as follows. A mixture of the ingredients is prepared, with the mixture blended long enough so as to avoid foaming and ensure proper hydration of the ingredients. The mixture is then pasteurized and homogenized according to standard techniques in the art. The homogenization can be done in one or two steps. The mixture is then cooled to a refrigeration temperature, which is typically about 2°C to about 8°C, and aged as needed, typically for a period of about 4 to about 24 hours. The steps of pasteurization, homogenization and cooling can be done in batch steps or continuously. After aging, the mixture is then rapidly frozen, typically at about -2°C to about -18°C to a product overrun of about 30% to about 120%. During freezing and any subsequent temperature cycling, gelation of the starch can occur. This frozen mixture is then hardened, typically to a temperature of about -20°C to about -40°C for a period of about 12 to about 48 hours.

In the examples that follow, all parts and percentages are given by weight and all temperatures in degrees Centigrade (°C) unless otherwise indicated. The following examples are presented to further illustrate and explain the present invention and should not be taken as

limiting in any regard. All percents used are on a weight/weight basis. The following analytical and testing procedures were used to characterize the starch products herein.

EXPERIMENTAL - PROCEDURAL

5 A. Starches Evaluated

Numerous starches were evaluated for effectiveness as to shape retention (*i.e.*, melt down), reduced iciness (*i.e.*, minimized growth of ice crystals), and reduced expansion/contraction due to changes in atmospheric pressure (*e.g.*, transporting at high altitude) in ice cream against a low fat (5%) control and a regular or full fat (10%) commercially available ice cream. Starches were also evaluated for their effectiveness as to the prevention of expansion and shrinkage of ice cream due to atmospheric pressure changes when ice cream is transported over high altitudes. Seventeen different illustrative starch samples are identified in Table I below

Table I – Description of Starches

ID #	Short Description	Starch Preparation
1	Native waxy corn starch	unmodified waxy corn starch
2	Acid converted waxy corn starch (20 WF)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 20 is obtained
3	Acid converted waxy corn starch (40 WF)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 40 is obtained
4	Acid converted waxy corn starch (80 WF)	waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 80 is obtained
5	Acid converted sago starch	sago starch treated with approx 0.7% HCl at 52 C until a approx 58.5WF is obtained
6	Native regular corn starch	unmodified regular corn starch
7	Acid converted sago starch treated with octenyl succinic anhydride	sago starch treated with approx 0.7% HCl at 52 C until a approx 58.5WF is obtained and further treated with OSA to obtain approx 1.75% bound OSA
8	Acid converted waxy corn starch (80 WF)	waxy corn starch treated with up to approx 3.0% HCl at 52 C until a desired WF up to 80 is obtained
9	Native waxy rice starch from Bangkok Starch Company	unmodified waxy rice starch
10	Waxy corn starch treated with propylene oxide and phosphorous oxychloride	waxy corn starch treated with PO to obtain approx 8.6% bound PO and further treated with approx 0.004% POCI ₃
11	Tapioca starch treated with propylene oxide and phosphorous oxychloride	Tapioca starch treated with PO to obtain approx 5.0% bound PO and further treated with approx 0.011% POCI ₃
12	Acid converted waxy corn starch treated with propylene oxide	Waxy corn starch treated with up to approx 1.0% HCl at 52 C until a desired WF up to 60 is obtained and further treated with PO to obtain approx 5.2% bound PO

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13	Acid converted sago starch treated with propylene oxide	Sago starch treated with approx 0.7% HCl at 52 C until a approx 58.5WF is obtained and further treated with PO to obtain approx 5.4% bound PO
14	Acid converted sago starch treated with sodium tripolyphosphate	Sago starch treated with approx 0.7% HCl at 52 C until a approx 58.5WF is obtained and further treated with STP to obtain approx 0.43% bound PO4
15	Taploca canary pyrodextrin	Taploca starch treated with anhydrous hydrogen chloride gas to a pH of approx 2.8 and further dry roasted to a maximum temperature of 154 C and held at temperature until approx 100% solubility and approx 2.2 ABF viscosity is obtained
16	Waxy corn starch enzymatically debranched	dispersed waxy corn starch treated with approx 6% Promozyme 400L (from Novo) until approx 85% short-chain-amylose is obtained and recovered by spray-drying
17	Acid converted potato starch (60 WF)	potato starch treated with up to approx. 2.0% sulfuric acid at 52 C until a desired WF up to 60 is obtained

B. Ice Cream Preparation

Three separate low fat (5%) ice cream formulations were prepared according to the formulation and procedure defined below –

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Table II – Formulation for Low Fat (5%) Ice Cream Mix

Ingredients	Control Formula (%)	2% Starch Formula (%)	4% Starch Formula (%)
Heavy Cream	13.10	13.10	13.10
Skim Milk	60.80	60.80	60.80
Skim Milk Powder (NFDM)	4.66	4.66	4.66
Sucrose	10.00	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.50	3.50
Corn Syrup Solids DE 24	5.50	4.50	3.50
Stabilizer/Emulsifier Blend	0.44	0.44	0.44
Starch	-	2.00	4.00
Total	100.00	100.00	100.00

Forty (40) percent of the total sucrose and the emulsifier/stabilizer blend were dispersed in the skim milk and mixed for five (5) minutes. A dry blend of NFDM, the remaining sucrose, corn syrup solids and the starch product to be evaluated (as required) was added and mixed for ten (10) minutes. The heavy cream was then added to the mixture, and the mixture gently blended so as to avoid foaming. With all ingredients blended together, the mixture was then pasteurized, homogenized and cooled to 4°C, respectively. Pasteurization was performed at 82°C for thirty (30) seconds, and homogenization was done in two stages, first at 17 MPa and then at

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3.4 MPa. Flavoring was then added to the ice cream. The ice cream mixture was aged overnight at 4°C. This mixture was then frozen in a continuous freezer (Technogel 100, available from Waukesha Cherry-Burrell, Delavan, Wisconsin) to an overrun of 100% by keeping the flow rate constant and adjusting the air supply. The mixture was filled into eight ounce (237 ml) sample cups, cooled to -20°C, and then hardened at -30°C for 24 hours. The resulting ice cream contains approximately 5% butterfat, 10% milk solids non-fat ("MSNF"), and 37% solids, based on a calculation using typical nutritional data. Each sample cup was subjected to heat shock treatment as described in the following Section C prior to further evaluation.

C. Heat Shock Stability Test

Temperature fluctuation in the distribution chain was simulated by exposing all ice cream samples in their containers to 12 temperature cycles in a freezer (model 34-25 commercially available from ScienTemp Corporation, Adrian, Michigan). The freezer was filled with one layer of 100 samples so that airflow could occur around all samples. Cycling conditions were -18°C for 12 hours followed by -6.7°C for twelve hours.

D. Melt Down Test

An ice cream sample was placed on a screen in a temperature-controlled cabinet at 19°C. A receptacle was placed on a balance below the screen to collect and weigh the meltdown or drip loss (m_d) over a time period of up to four (4) hours. The initial weight of the ice cream (m_0) was determined and kept within a ten percent (10%) standard deviation. Weight of the drip loss (m_d) was recorded over the four-hour time period. Total melt down (MD, %) was determined at the end of the four-hour period according to the following equation -

$$MD = \frac{m_d}{m_0} \times 100$$

E. Sensory Evaluation

Samples were evaluated using the descriptive analysis method with a 15-point scale. During each session 2 test samples were evaluated by an expert panel for 10 different attributes as listed and defined below. A control sample that had gone through temperature cycling was used as a base reference and presented with the samples. Each test sample was evaluated twice to reduce the standard deviation.

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Definitions of descriptive analysis:

1. "Descriptive analysis provides quantitative descriptions of products, based on perception of a group of trained subjects. The description takes into account all sensations that are perceived when a product is evaluated." (On-line training course, U.C. Davis, The Regents of the University of California and Dr. Jean-Xavier Guinard, 2004).
2. "All descriptive analysis methods involve the detection (discrimination) and the description of both the qualitative and quantitative sensory aspect of a product by trained panels of 5-100 judges (subjects).....These qualitative factors include terms which define the sensory profile or picture or thumbprint of the sample.....The intensity or quantitative aspect of a descriptive analysis expresses the degree to which each of the characteristics is present. This is expressed by the assignment of some value along a measurement scale." ("Sensory evaluation techniques" M.Meilgaard, G.V. Civille, B.T.Carr, 3rd edition 1999).

3. DESCRIPTORS

- | | |
|----------------------------|---|
| Firmness - | Force required to compress the sample with teeth and tongue. |
| Coldness - | Tendency to cool surfaces of the mouth. |
| Cohesiveness - | Tendency to resist loss of structure during chewing. A cohesive product is elastic and maintains structure during chewing. A non-cohesive product is brittle, easily fragments or is crumbly in texture, and provides a clean bite through. |
| Rate of Breakdown - | Rate at which a product softens or fragments upon chewing. |
| Ice crystals - | Perception of 'grittiness' caused by the number and size of ice crystals. Ideally, no ice crystals can be detected in the mouth. |
| Melt Thickness - | Viscosity of film left on the palate. |
| Smoothness - | Tendency of the film on the palate to be smooth, slick and non-sticky. |
| Gumminess - | Residual film in the mouth is tacky, sticking the tongue to the surfaces of the mouth. |
| Vanilla Flavor - | Authentic note of vanilla and cream. |
| Off-Note - | Any inappropriate aroma. |

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F. Rheological Testing Procedure

Each of the ice cream mixes collected after the homogenization step in Section B above was poured into molds and held in the freezer at -18°C . One mold of each formulation was removed from the freezer and tested on the rheometer as soon as it equilibrated to 25°C .
5 Another mold was removed from the freezer and placed in a constant temperature bath at -6.7°C where it was held for 7-8 days, before it was removed, equilibrated to 25°C and tested on the rheometer.

A sequence of rheology experiments was performed on each sample as described in Table III below -

10 **Table III - Rheology Tests Run on Each Sample**

Test Name	Specific Conditions
Dynamic Strain Sweep	$\omega=1\text{ rad/s}$, $\gamma_{\text{min}}=.1\%$, $\gamma_{\text{max}}=100\%$
Dynamic Frequency Sweep	$\gamma < \gamma_{\text{cr}}$, $\omega_{\text{min}}=1\text{ rad s}^{-1}$, $\omega_{\text{max}}=100\text{ rad s}^{-1}$
Steady Shear Step Rate	Shear rate= 1 s^{-1} , length of experiment= 120 s
Steady Shear Rate Sweep	Time per shear rate= 30 s , shear rate range= $1-100\text{ s}^{-1}$

In Table III, "min" refers to minimum, "max" refers to maximum, " ω " refers to frequency, " γ " refers to strain, " γ_{cr} " refers to critical strain, "rad" refers to radians, and "s" refers to seconds. Rheological tests described above are according to standard rheological terms.

15 Experiments on the ice cream mixes in the molds were all done at 25°C . The data tabulated from these experiments includes: from the dynamic frequency sweep test, the elastic modulus (G' , Pascal) at 10 rad/s and $\tan \delta$ at 10 rad/s ; from the dynamic strain sweep, γ_{cr} and the peak in steady shear viscosity; from the shear step rate, viscosity (η , Pascal-sec) at $1/\text{s}$, η after 120 s at $1/\text{s}$ and the final η after 120 s at $1/\text{s}$; and the steady shear rate sweep, or η at $100/\text{s}$ (Pascal-
20 sec).

G. Differential Scanning Calorimetry Procedure for Determining T_g

Differential scanning calorimetry measurements were performed in a Perkin-Elmer DSC-7 with CCA cooling system (Norwalk, Connecticut). Liquid N_2 was used for sub-ambient
25 temperature experiment. The instrument was calibrated with indium and water. An empty stainless-steel pan was used as a reference.

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Approximately 10 mg of starch (dry base) was weighed into large volume stainless steel pan. Extra water was added to the sample to reach the desired moisture content. Each starch was tested at two solid levels (40% and 20%) using quench cooling and annealing method to ensure that the measured T_g' and W_g' values are close to real values. The starch/water mixture was first heated from 10°C to 160°C at 10°C/min to ensure that all the starch was fully dispersed in water. Then the starch/water mixture was quench cooled to -70°C and was held for 15 minutes to ensure maximum amount of ice formed. The sample was then heated at 2.5°C/min. to detect the T_g' and the amount of the frozen water. To maximize the amount of ice formed in the ice cream mix, the samples were also subjected to an annealing procedure before temperature scanning. The sample was held at the temperature slightly above T_g' for 30 minutes and then was cooled to below T_g' at 1.0°C/min and held at -40°C for another 10 minutes. The samples were then heated at 2.5°C/min to 20°C. The middle point of the glass transition was reported as T_g' , and the ice melting enthalpy was used to calculate the W_g' value of the starch. Duplicate tests were run for each solid level or cooling condition. The average values from those tests that generated maximum amount of ice were reported.

H. Altitude Test

Ice cream samples were collected in 400ml stainless steel custom made cups. The cups were overfilled, hardened for 24 hours at -30°C according to the standard procedure and tested the next day (without heat shock treatment). Before the test the ice cream in each cup was leveled off and the cup was placed in a desiccator attached to a vacuum pump. This was stored in a Styrofoam box with dry ice in order to maintain the temperature throughout the test. The test was performed at a temperature of -18°C and a pressure of -67.73 kPa (-20 inch Hg) for 15 minutes. The expansion was measured after 15 minutes and is expressed in mm or illustrated in a photo. After the test the samples were kept in a freezer at -18°C for 24 hours. Shrinkage was then determined by measuring the amount of glycol used to fill the gap between the ice cream and the cup (reported in g glycol). Two repeats were run in order to ensure reproducibility.

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EXPERIMENTAL - RESULTS

1. Melt Down and Sensory Results

5 The effect of the starch according to the present invention on the melt down on ice cream is shown in Figure 1. From Figure 1 it is seen that unmodified or lightly converted starches are preferred. From Figure 1 it is also seen that waxy starches are preferred as the base starch, more preferably, waxy corn starches. Additionally, it can be seen that certain amylose-containing starches provide effective reduction of melt down.

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Table IV – Meltdown/Ice Crystal Data on Ice Creams with Starch

Sample ID	Meltdown%	Ice Crystals
Starch 16 at 2%	43.00	5.95
Starch 16 at 4%	23.93	4.45
Starch 2 at 2%	16.00	4.7
Starch 3 at 2%	21.00	4.85
Starch 4 at 2%	37.00	4.75
Starch 4 at 4%	4.00	2.65
Starch 8 at 2%	44.00	4.8
Control ice cream without starch	69.00	7.5
Starch 12 at 2%	79.00	6.45
Starch 12 at 4%	97.00	3.45
Starch 14 at 2%	25.00	4.8
Starch 13 at 2%	86.00	5.15
Starch 7 at 2%	49.00	4.85
Starch 1 at 2%	14.37	4.7
Starch 6 at 2%	19.73	5.45
Starch 9 at 2%	86.18	5.35
Starch 10 at 2%	80.09	5.1
Starch 5 at 2%	37.10	6.7
Commercial 10% fat ice cream	44.00	4.25

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Those skilled in the art recognize that certain processing conditions (e.g., reduced overrun) and variations in the formula (e.g., higher fat content) can result in improved melt down. The present invention shows that the addition of certain starches improves meltdown, even more than presently used emulsions and stabilizers. Further, Table IV illustrates that certain starches provide an improved reduction in meltdown of a frozen dessert (e.g., starches 2-4, 8 and 16) versus frozen desserts formulated without the functional starch.

2. Rheological Testing Results

The ice cream mixes show significant changes in rheology during storage at -6.7°C . For most of the samples, $\tan \delta$ decreases over time, which indicates the development of gelled structures under frozen conditions. Trends are similar for low-fat and non-fat formulas. After 7-10 days, the least gelled (highest $\tan \delta$) samples are Sample 10 and Sample 12 while the most gelled (lowest $\tan \delta$) samples are Sample 1, Sample 2 and Sample 6. The viscosity of each mix increased or stayed constant throughout the time that the samples were held at -6.7°C . The modulus, G' , showed similar behavior to the viscosity, η .

Table V – Rheology Data on Ice Cream Mixes with Starch

Sample	$\tan \delta$ at 10 rad/s fresh	η at 1/s fresh	G' at 10 rad/s fresh	$\tan \delta$ at 10 rad/s @ 7-8 days	η at 1/s @ 7-8 days	G' at 10 rad/s @ 7-8 days
Starch 16 at 2%	1.5724	0.2476	0.7279	0.8978	0.9250	3.2386
Starch 16 at 4%	1.2644	0.4205	1.4463	0.472	3.8639	22.018
Starch 2 at 2%	1.6801	0.3120	1.6682	0.2538	15.2720	193.6
Starch 3 at 2%	2.0387	0.2614	0.9887	0.3665	7.9273	52.157
Starch 4 at 2%	2.2618	0.2089	0.6088	0.3597	6.4949	48.769
Starch 4 at 4%	1.8449	0.3314	1.5129	0.3263	46.5130	865.84
Starch 8 at 2%	1.9657	0.2037	0.5129	0.7069	2.8037	12.249
Control ice cream without starch	1.7367	0.1982	0.5058	1.6172	0.3059	0.9667
Starch 12 at 2%	2.2580	0.2277	0.686	1.93705	0.2699	0.9439
Starch 12 at 4%	2.0553	0.3717	1.4746	1.6779	0.5526	2.5249
Starch 14 at 2%	0.9583	1.2793	3.9336	0.36985	12.4740	91.049
Starch 13 at 2%	2.1291	0.2271	0.6886	1.3858	0.5244	1.9084
Starch 7 at 2%	1.3449	0.5992	1.7108	0.46385	4.7607	20.386
Starch 1 at 2%	1.9E+00	5.1E-01	2.0E+00	0.31	1.2E+01	8.8E+01
Starch 6 at 2%	7.2E-01	2.6E+00	9.5E+00	0.31	1.8E+01	1.3E+02
Starch 9 at 2%	2.1E+00	4.4E-01	1.8E+00	1.4	7.6E-01	3.1E+00
Starch 10 at 2%	2.1E+00	7.0E-01	2.5E+00	1.5	9.2E-01	4.2E+00
Starch 5 at 2%	1.5E+00	5.2E-01	1.5E+00	0.6	1.8E+00	5.8E+00

The correlation between the reduced melt down of ice cream and the ability of the starch to gel in the model ice cream system under freezer conditions is shown in Figure 2. As can be seen from Figure 2, a starch having a $\tan \delta$ of less than about 1.0 results in a frozen dessert having a melt down of about fifty percent (50%) or less. Starches having a $\tan \delta$ of less than about 0.4 resulted in an ice cream with the slowest melt down.

As previously noted, the results of the effectiveness of the above samples with respect to melt down are illustrated in Figure 1. Those frozen desserts containing about two to about four

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percent of a starch that gels during freezing, particularly those starches having a $\tan \delta$ less than about one, resulted in reduced melt down and improved shape retention after exposure to heat shock.

5 3. Tg' Results

Those starches identified as providing good ice crystal growth inhibition capability included native starches, acid or enzyme converted starches, or chemically modified versions of those starches having a DE less than about 8, and a Tg' greater than about -6°C (Tg' > ~ -6°C) and, water binding property of Wg' greater than about 0.30 (Wg' > ~ 0.30) (unfreezable water weight fraction in the unfrozen glass state). Tg' and Wg' are determined by the calorimetry method described above. The results are provided in Table VI below –

Table VI

Starch Sample	Tg' (°C)	Wg'(g/g total glass)	Iciness
Starch 1	-4.521	0.309	4.70
Starch 4	-5.627	0.352	4.00
Starch 8	-5.321	0.325	4.60
Starch 11	-5.993	0.308	4.70
Starch 7	-5.127	0.310	4.85
Starch 14	-5.550	0.318	4.80
Starch 12	-7.766	0.322	6.45
Starch 13	-8.370	0.257	5.30
Starch 16	-7.021	0.300	5.95
Starch 15	-8.651	0.269	5.10

In the present invention, corn syrup solids were reduced to keep the total solids content of the formulation constant. Adding starch to replace fat can affect the glass transition temperature, freezing temperature and the 'water binding' property of the serum phase in the frozen state. Starches were compared at 2% and 4% usage levels. At about 3% to about 4% usage level, the starches of the present invention function in the absence of other stabilizers, providing improved heat shock stability characterized by a reduction in ice crystals as compared to a non-starch control (see Section 4 below). Higher levels of starch generally further improve performance with respect to melt down and heat shock stability. However, above about 4%, negative textural attributes occur.

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4. Low Fat Ice Cream with Starch

Ice cream formulations were prepared according to the formulation defined in Table VII below –

5 Table VII - Ice cream formulation (5% butterfat, 3% starch 4, no other stabilizer)

Ingredients	Control Formula (%)	3% Starch Formula (%)
Heavy Cream	13.10	13.10
Skim Milk	60.80	60.80
Skim Milk Powder (NFDm)	4.66	4.66
Sucrose	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.15
Corn Syrup Solids DE 24	5.50	4.15
Emulsifier/Stabilizer Blend	0.44	-
Emulsifier	-	0.14
Starch 4	-	3.00
Total	100.00	100.00

As in the 'Ice Cream Preparation' section above, when starches of the present invention were added, the 42DE corn syrup solids and the 24DE corn syrup solids were reduced to keep the total solids constant. However, here the stabilizer was also omitted. As noted in Section 3 *supra*, the starches of the present invention function in the absence of other stabilizers. Each ice cream mix was prepared according to the procedure described in the 'Ice Cream Preparation' section.

Figure 3 is illustrative of a frozen dessert made with 3% starch according to the formula described in Table VII above, *i.e.*, without any stabilizer. This frozen dessert was subjected to the heat shock test described in Section C with the exception that the ice cream was cycled out of the container. From Figure 3 it is seen that the heat shock treated frozen dessert with 3% starch retained its shape. Figure 4 quantitatively illustrates the relationship between meltdown and shape retention over time.

5. Non-fat Ice Cream Mix with Starch and Stabilizer

20 Ice cream formulations were prepared according to the formulation defined in Table VIII below –

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Table VIII – Rheological Ice Cream Formulation

Ingredients	1
	0% Fat
Heavy Cream	0.00
Skim Milk	72.67
Non-Fat Dry Milk	4.82
Sucrose	10.50
42DE Corn Syrup Solids	5.78
24DE Corn Syrup Solids	5.78
CREST Stabilizer/Emulsifier	0.46

As in the 'Ice Cream Preparation' section above, when starches of the present invention were added, the 42DE corn syrup solids and the 24DE corn syrup solids were reduced equally to keep the total solids constant. Each ice cream mix was prepared according to the procedure described in the 'Ice Cream Preparation' section. Seven starches were evaluated in the non-fat formula at 2.1% wt/wt.

The ice cream mixes show significant changes in rheology during storage at -6.7°C. For most of the samples, tan δ decreases over time, which indicates the development of gelled structures under frozen conditions. After 7-10 days, the least gelled (highest tan δ) sample is Sample 10 while the most gelled (lowest tan δ) samples are Sample 1 and Sample 6. The viscosity of each mix increased or stayed constant throughout the time that the samples were held at -6.7°C. The modulus, G', showed similar behavior to the viscosity.

Table IX – Rheology Data on Non-fat Ice Cream Mixes

Sample	tan δ at 10 rad/s fresh	η at 1/s fresh	G' at 10 rad/s fresh	tan δ at 10 rad/s @ 7-8 days	η at 1/s 7-8 days	G' at 10 rad/s 7-8 days
Starch 1 at 2.1%	3.9E+00	2.1E-01	4.4E-01	2.6E-01	1.5E+01	1.0E+02
Starch 6 at 2.1%	9.0E-01	1.3E+00	4.2E+00	3.1E-01	8.0E+00	7.4E+01
Starch 10 at 2.1%	3.1E+00	3.2E-01	7.8E-01	2.1E+00	5.0E-01	1.5E+00
Starch 4 at 2.1%	4.0E+00	5.8E-02	1.2E-01	3.8E-01	7.8E+00	4.6E+01
Starch 5 at 2.1%	1.5E+00	4.1E-01	8.4E-01	3.9E-01	2.6E+00	1.4E+01

The data further shows that, while frozen dessert mixes prepared with functional amylose-containing starches have a drop in tan δ that occurs quickly after freezing, waxy starches drop more gradually in tan δ when held at frozen conditions for extended periods. This illustrates that functional amylose-containing starches are preferred over waxy starches for use in frozen desserts that are not subject to extensive temperature cycling.

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6. Altitude Testing

Ice cream samples were prepared according to the formulation defined in Table X below

Table X - Ice cream formulation (5% butterfat, 3% starch 4 or 17, no other stabilizer)

Ingredients	Control Formula (%)	3% Starch Formula (%)
Heavy Cream	13.10	13.10
Skim Milk	60.80	60.80
Skim Milk Powder (NFDM)	4.66	4.66
Sucrose	10.00	10.00
Corn Syrup Solids DE 42	5.50	4.15
Corn Syrup Solids DE 24	5.50	4.15
Emulsifier/Stabilizer Blend	0.44	-
Emulsifier	-	0.14
Starch	-	3.00
Total	100.00	100.00

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The samples were tested according to Experimental Procedure H described above. Table XI below shows that samples containing 3% starch show a reduced amount of shrinkage of the ice cream when exposed to a defined vacuum. This is further illustrated in Figure 5. While the control in Figure 5 shows significant expansion, the starch containing samples do not expand under these test conditions. Trials in a higher fat formula and in the presence of other stabilizers or the evaluation after heat shock provided similar results.

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Table XI - Shrinkage results of ice cream expressed in g glycol

Sample	Initial Weight (g)	Final Weight (g)	Glycol Added (g)
Control	351.4	390.7	39.3
Control	378.0	415.9	37.9
3% Starch 4	370.8	393.5	22.7
3% Starch 4	331.9	357.0	25.1
3% Starch 17	311.6	325.4	13.8
3% Starch 17	366.8	386.4	19.6

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From the above Experimental results it is seen that certain starches function better than others. For example, with respect to shape retention and changes in altitude, useful starches include amylopectin starches such as waxy corn, waxy tapioca or waxy potato. These starches can be used in their native form, crosslinked, and/or lightly converted (e.g., up to about 80 WF).

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Other useful starches include amylose-containing starches from a variety of bases such as sago,

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tapioca, potato, corn or rice. These amylose-containing starches can be in their native form or lightly converted (e.g., up to about 80 WF).

For reducing iciness, useful starches include amylopectin starches that are native or lightly converted (e.g., up to about 80 WF); amylose-containing starches from a variety of bases
5 such as sago, tapioca, potato, corn or rice in their native form or lightly converted (e.g., up to about 80 WF); and amylose and amylopectin starches that are crosslinked and/or lightly stabilized. The degree and balance of crosslinking and stabilization can be determined so that it meets the Tg' and Wg' criteria.

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Although the present invention has been described and illustrated in detail, it is to be understood that the same is by way of illustration and example only, and is not to be taken as a limitation. The spirit and scope of the present invention are to be limited only by the terms of any claims presented hereafter.

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What is claimed and desired to be secured by Letters Patent is:

1. A frozen dessert comprising a functional starch able to gel during freezing conditions, wherein the frozen dessert has a $\tan \delta$ of about 1.0 or less.
2. The frozen dessert of claim 1 comprising a $\tan \delta$ of about 0.4 or less.
3. The frozen dessert of claim 1 wherein the starch is a converted starch having water fluidity ("WF") of from about 0 to about 80.
4. The frozen dessert of claim 3 wherein the starch has been converted to WF of from about 20 to about 65.
5. The frozen dessert of claim 3 wherein the converted starch is a waxy starch.
6. The frozen dessert of claim 3 wherein the converted starch is a potato starch.
7. The frozen dessert of claim 1 wherein the starch is an amylopectin starch.
8. The frozen dessert of claim 7 wherein the amylopectin starch is waxy corn.
9. The frozen dessert of claim 1 wherein the functional starch provides a reduction in the meltdown of the frozen dessert versus frozen desserts prepared without the functional starch.
10. The frozen dessert of claim 1 wherein the frozen dessert is a novelty frozen dessert and the starch provides retention in the shape of the frozen dessert when the novelty frozen dessert is heat shock treated for up to 12 freeze/thaw cycles, each freeze/thaw cycle lasting up to 24 hours.

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11. The frozen dessert of claim 1 wherein the starch provides the frozen dessert a reduced amount of shrinkage of the ice cream when exposed to a defined vacuum as compared to a frozen dessert without the starch.
12. The frozen dessert of claim 1 wherein the frozen dessert is selected from the group consisting of ice cream, ice milk, water ice and parfaits.
13. The frozen dessert of claim 12 wherein the frozen dessert is ice cream.
14. The frozen dessert of claim 13 wherein the ice cream is low fat ice cream.
15. A frozen dessert comprising a functional starch having a glass transition temperature (T_g) of about -6°C or greater, a water binding property (W_g) of about 0.30 g/g or greater, wherein the functional starch provides improved ice crystal inhibition in a frozen dessert versus a frozen dessert prepared without the functional starch.
16. A process for preparing a frozen confectionery, the process comprising the steps of:
preparing a mixture of ingredients for the frozen confectionery, the mixture including a functional starch able to gel during freezing conditions,
wherein the mixture has a $\tan \delta$ of about 1.0 or less,
freezing the mixture within a temperature range of from about -2°C to about -8°C ; and
wherein the step of freezing effects gelling of the starch, and
wherein the frozen confectionery has an improvement in melt down over frozen confectionery that do not have the starch as an ingredient.
17. The process according to claim 16 wherein the starch is an amylopectin starch.
18. The process according to claim 17 wherein the amylopectin starch is a waxy corn.
19. The process according to claim 16 further comprising the step of pasteurizing the mixture within a temperature range of from about 70°C to about 100°C .

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20. The process according to claim 16 further comprising the step of homogenizing the mixture within a pressure range of from about 2MPa to about 20 MPa.
21. The process according to claim 16 further comprising the step of cooling the mixture within a temperature range of from about 2°C to about 8°C.
22. The process according to claim 16 further comprising the step of aging the mixture for a time period of about 4 hours to about 24 hours.
23. The process according to claim 16 further comprising the step of hardening the mixture at a temperature of from about -20°C to about -40°C.
24. The process according to claim 16 further comprising the step of replacing at least part of solids ingredients of the frozen confectionery with the starch.
25. A functional starch for use in frozen desserts, the starch providing the frozen dessert starch a $\tan \delta$ of about 1.0 or less when formulated in the frozen dessert,
wherein the functional starch is able to gel during freezing conditions, and
wherein the functional starch provides improved meltdown, reduced iciness and/or reduced expansion/contraction of the frozen dessert versus a frozen dessert prepared without the functional starch.

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ABSTRACT

Use of starch(es) and starch derivatives in frozen desserts for improved meltdown and shape retention, including reduced expansion and contraction of the frozen dessert when transported at high altitudes, e.g., over mountain ranges. These improved characteristics are retained in the frozen dessert even after multiple heat shock cycling. The starch(es) and starch derivatives inhibit ice crystal formation in frozen after heat shock cycling.

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DECLARATION FOR PATENT APPLICATION

☒ Original ☐ Supplemental ☐ Substitute ☐ PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below), or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

STARCH FOR FROZEN DESSERTS
(Title of the Invention)

the specification of which (check one)

- ☒ Is attached hereto.
- ☐ was filed on _____ as Application Serial No. _____.
- ☐ Was described and claimed in PCT International Application No. – filed on – and as amended under PCT Article 19 on – (if any).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information that is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 (a) - (d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified, by checking the box below, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Applications			Priority Claimed		Copy Attached	
Application Number	Country	Foreign Filing Date (MM/DD/YYYY)	YES	NO	YES	NO

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below and claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT international application(s) designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

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Parent Application Number	Filing Date (MM/DD/YYYY)	Status (Mark Appropriate Column Below)		
		Patented	Pending	Abandoned
60/498,834	8/29/2003		X	
60/511,765	10/16/2003		X	

As a named inventor, I hereby revoke all prior powers and appoint those attorney(s) and/or agent(s) at Customer Number 35157 to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

I acknowledge the above-indicated attorneys and agents represent my employer (if I am an employee and this application has been or will be assigned to my employer) or the entity with which I have contracted (if I am an independent contractor and this application has been or will be assigned to such entity) and in such cases do not represent me individually. I further acknowledge I have not established, nor will I seek to establish, any personal attorney/client relationship with the above named attorneys in connection with this application and understand that, should I require legal representation, I will obtain such, at my expense, other than through the above indicated attorneys.

Send Correspondence to: ☒ Customer Number 35157

OR ☐ NATIONAL STARCH AND CHEMICAL COMPANY
Post Office Box 6500
10 Finderns Avenue
Bridgewater, New Jersey 08807-3300

Direct telephone calls to: David P. LeCroy at (908) 685-5433

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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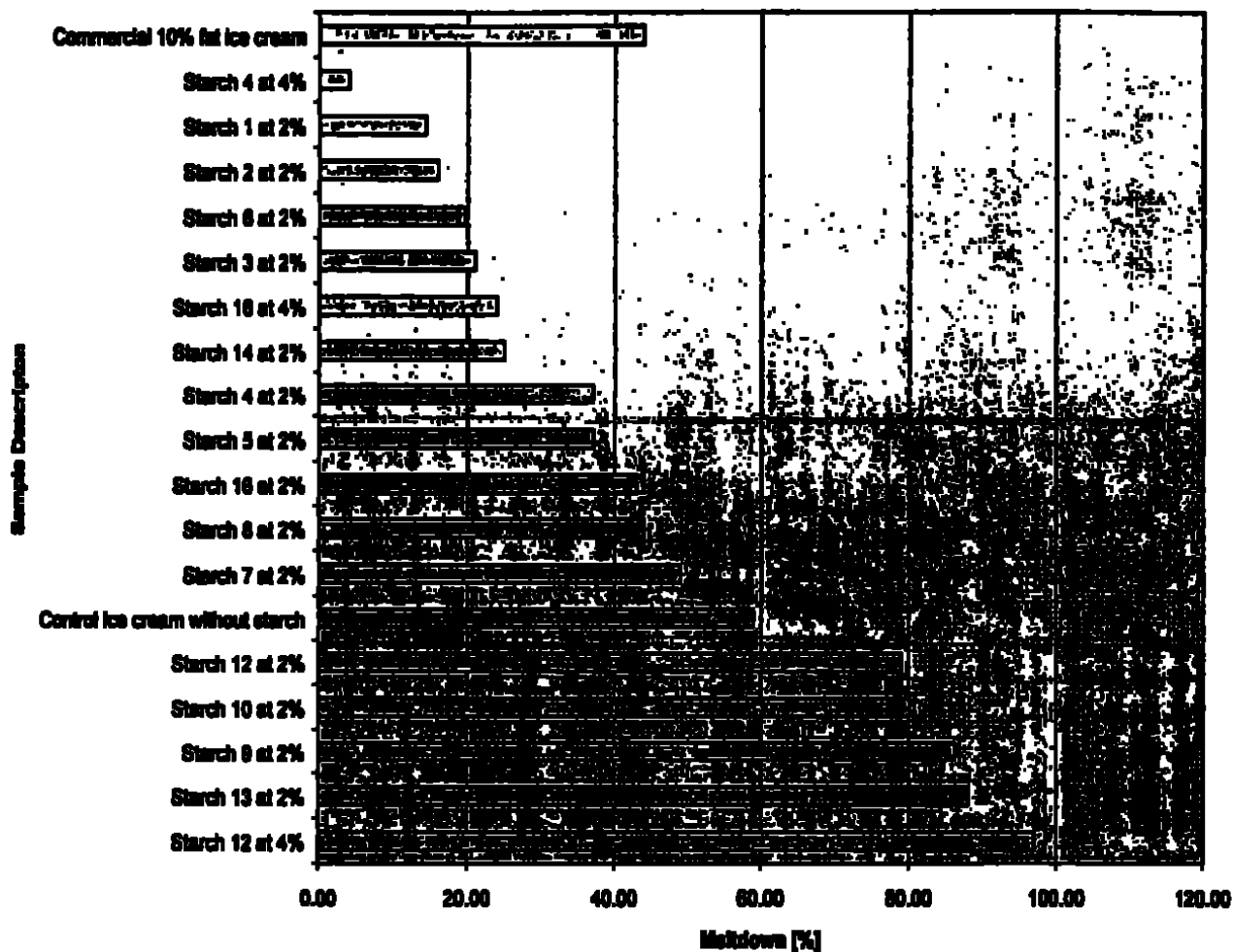


Figure 1. Graph illustrating percent melt down of various ice creams after 4 hours at 19°C

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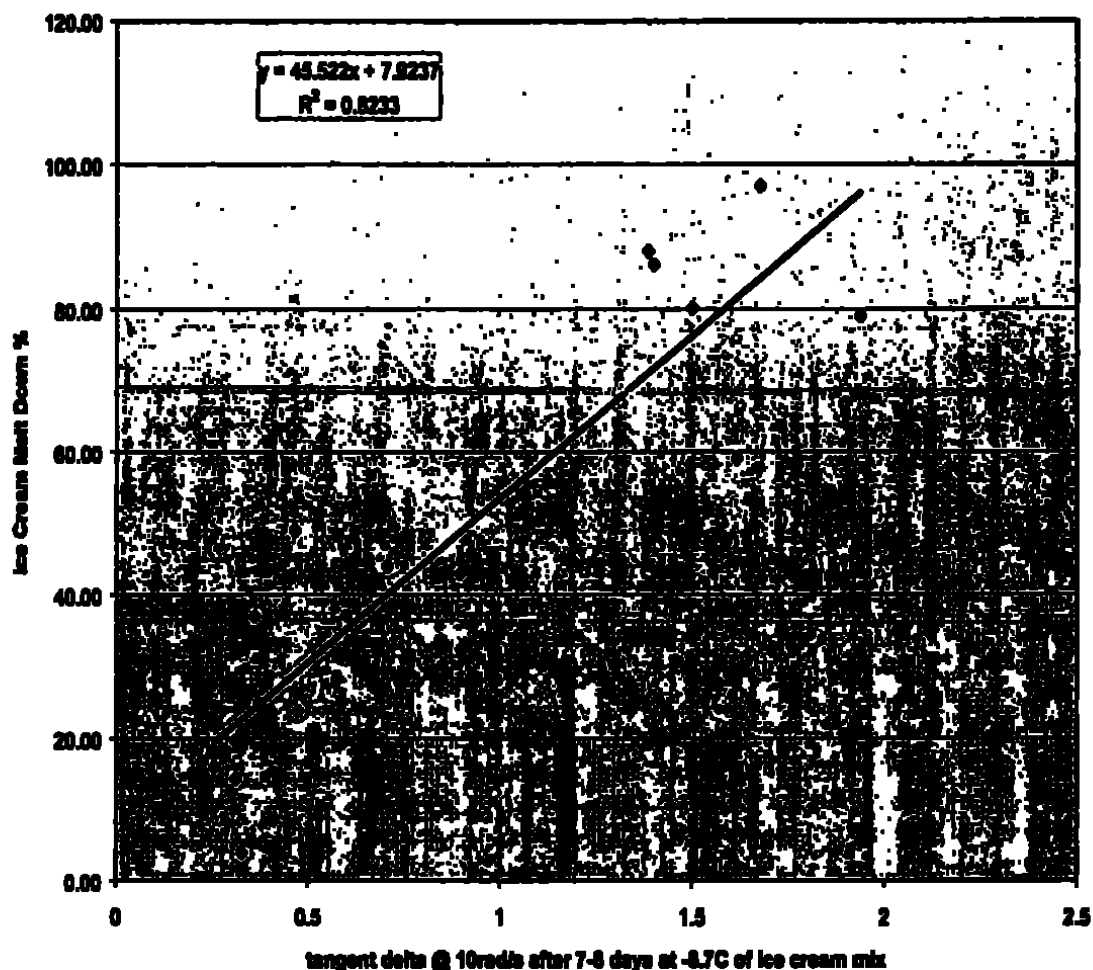


Figure 2. Graph illustrating correlation between melt down and $\tan \delta$
(starches provided in Table IV)

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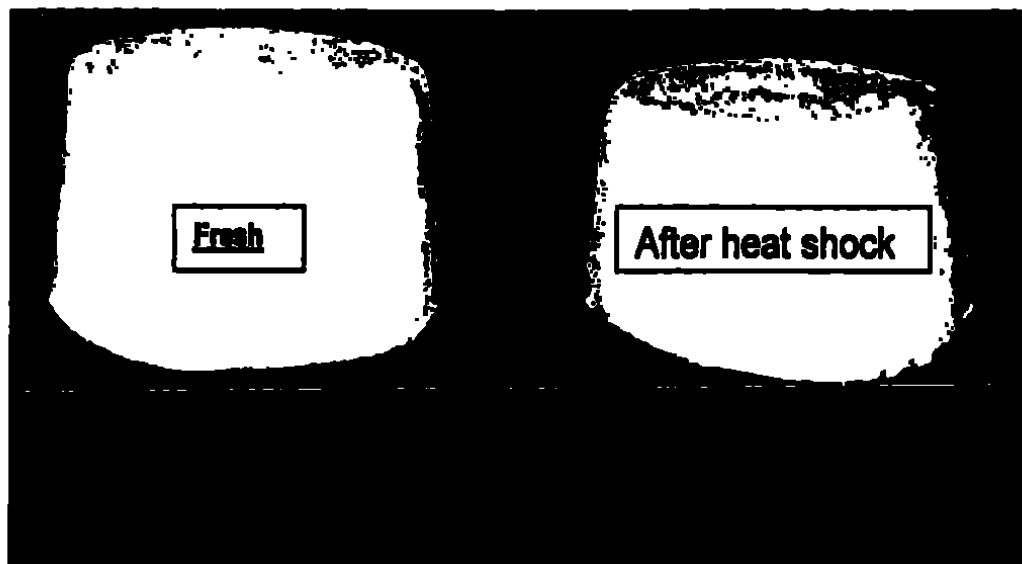


Figure 3. Photo of fresh and heat shocked ice cream containing 3% starch

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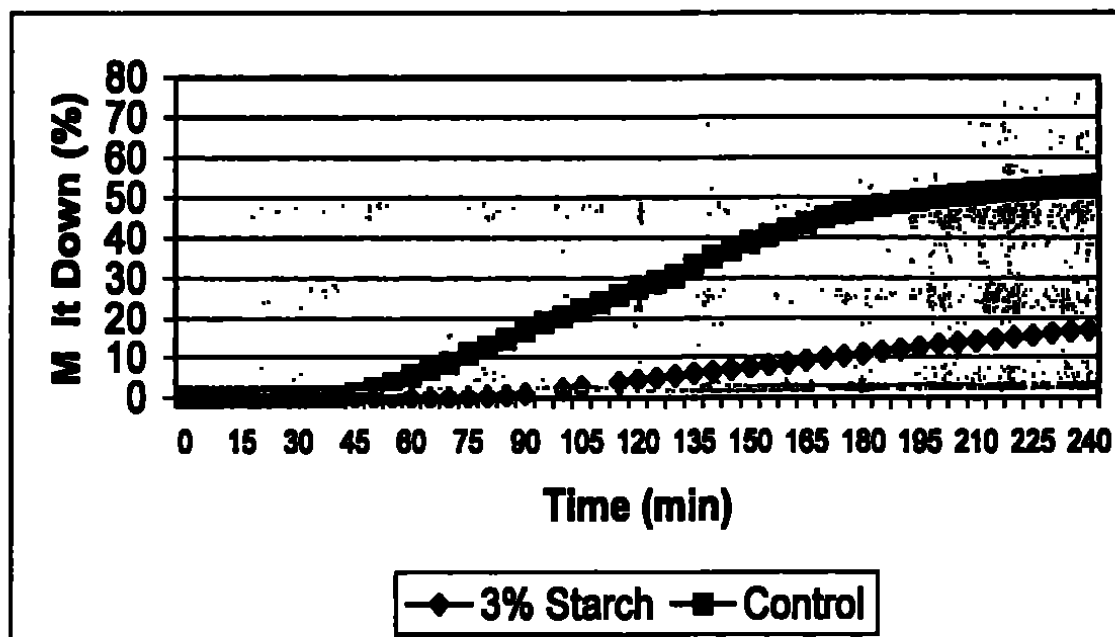


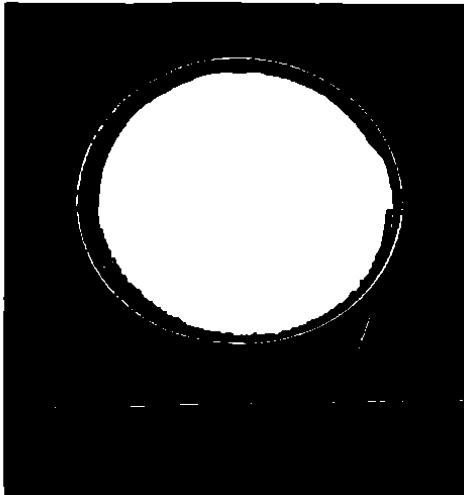
Figure 4. Melt curve of ice cream containing 3% starch

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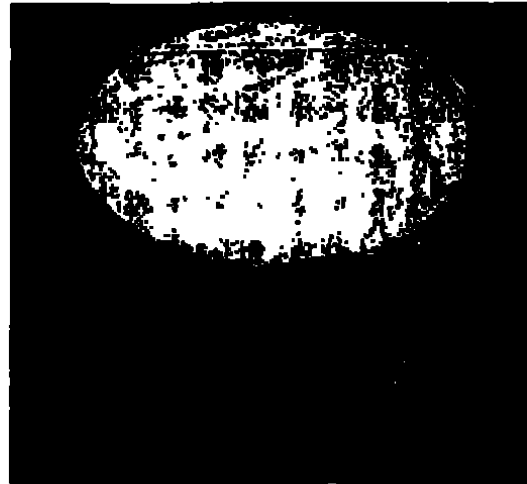
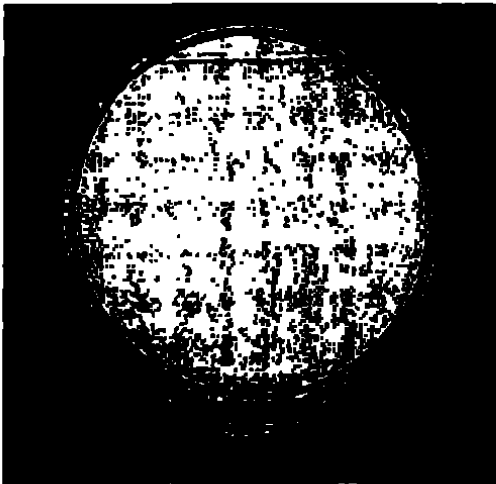
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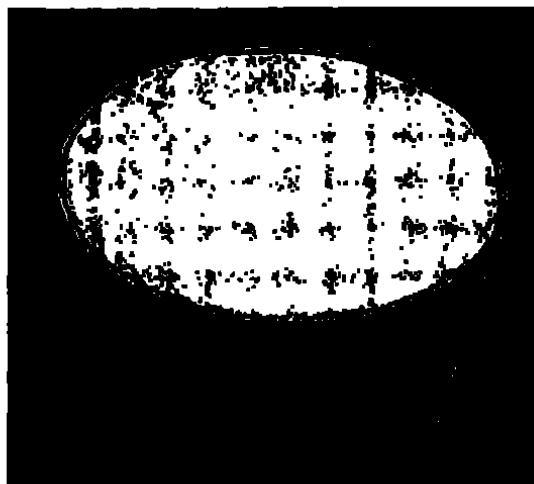
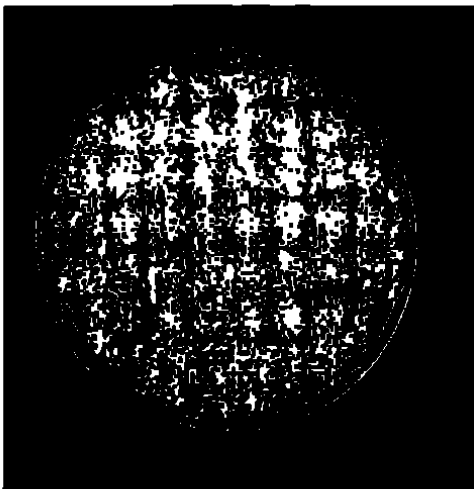
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Starch #4: Shrinkage Control Formula Expansion



Starch #17: Shrinkage 3% Starch #4 Expansion



Shrinkage 3% Starch #17 Expansion

Figure 5. Photos of ice cream without starch and ice cream containing 3% starch

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Certificado de Idoneidad Profesional No. 0045 expedido por la Universidad Nacional de Colombia, por el
Ministerio de Relaciones Exteriores y autorizado por el Ministerio del interior y de Justicia, por resolución
número 718 de 2003.

(Se traduce el Certificado de la Oficina de Patentes y Marcas de los EE.UU. de una Solicitud de Registro
de Patente No10/828,555 a favor de NATIONAL STARCH AND CHEMICAL COMPANY, y las
Reivindicaciones, escritos en Inglés.)

ESTADOS UNIDOS DE AMÉRICA

A TODOS A QUIENES ESTOS PRESENTES LLEGAREN:

DEPARTAMENTO DE COMERCIO DE LOS ESTADOS UNIDOS

OFICINA DE PATENTES Y MARCAS DE LOS ESTADOS UNIDOS

Noviembre 02, 2004

POR EL PRESENTE CERTIFICO QUE ADJUNTA SE ENCUENTRA UNA
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CON LOS REQUISITOS PARA QUE LE FUESE OTORGADA UN NÚMERO DE
PRESENTACIÓN BAJO EL TÍTULO 35 DEL CÓDIGO DE LOS ESTADOS
UNIDOS SECCIÓN 111.

SOLICITUD DE SOLICITUD: **10/828,555**

FECHA DE PRESENTACIÓN: **Agosto 26, 2004**

Por autoridad del

COMISARIO DE PATENTES Y MARCAS

(Firmado) M. SIAS, Funcionario que Certifica

(SELLO DE OBLEA EN RELIEVE DE LA OFICINA DE PATENTES Y MARCAS
DE LOS ESTADOS UNIDOS DE AMÉRICA)

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Lo que se reivindica y para lo cual se desea obtener protección mediante el Título de Patente es lo siguiente:

1. Un postre congelado que incluye un almidón funcional que se puede convertir en gel durante las condiciones de congelamiento, donde el postre congelado tiene una δ "tan" de 1.00 o menor.
2. El postre congelado de la reivindicación 1, que comprende una δ "tan" de 0.4 o menor
3. El postre congelado de la reivindicación 1, donde el almidón es un almidón convertido con fluidez de agua ("FA") de cerca de 0 a cerca de 80.
4. El postre congelado de la reivindicación 3, donde el almidón se ha convertido a una FA de cerca de 20 a cerca de 65.
5. El postre congelado de la reivindicación 3 donde el almidón convertido es una almidón ceroso.
6. El postre congelado de la reivindicación 3 donde el almidón convertido es una almidón de papa.
7. El postre congelado de la reivindicación, donde el almidón es un almidón amilopectina.
8. El postre congelado de la reivindicación 7 donde el almidón amilopectina es un maíz ceroso.
9. El postre congelado de la reivindicación 1, donde el almidón funcional, suministra una reducción del derretimiento del postre congelado comparado a postres preparados sin el almidón funcional.

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10. El postre congelado de la reivindicación 1, donde el postre congelado es un postre congelado novedoso y el almidón suministra la retención en la forma del postre congelado, cuando el postre congelado novedoso se somete a tratamiento térmico de choque, hasta por 12 ciclos de congelamiento / licuación, cada ciclo de congelamiento / licuación con una duración de hasta 24 horas.

11. El postre congelado de la reivindicación 1, donde el almidón le suministra al postre congelado una cantidad reducida de encogimiento del helado, cuando es expuesto a un vacío definido, comparado con un postre congelado sin el almidón.

12. El postre congelado de la reivindicación 1, donde el postre congelado se selecciona del grupo consistente en helado, leche congelada y postres hechos a base de varias clases de crema helada.

13. El postre congelado de la reivindicación 12, donde el postre congelado es helado.

14. El postre congelado de la reivindicación 12, donde el postre congelado es helado dietético (bajo en grasa).

15. Un postre congelado que comprende un almidón funcional con una temperatura de transición vítrea (T_g ') de cerca de -6° C o mayor, o una propiedad ligante de agua (Wg ') de cerca de 0.30 g/g o mayor, donde el almidón funcional suministra inhibición mejorada de cristales de hielo en un postre congelado comparado con un postre congelado preparado sin el almidón funcional.

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16. Un proceso para preparar confitería congelada, dicho proceso comprendiendo los siguientes pasos:

preparación de una mezcla de ingredientes para la confitería congelada, la mezcla incluyendo un almidón funcional capaz de convertirse en gel durante las condiciones de congelamiento,

donde la mezcla tiene una $\delta^{\circ}\text{tan}$ de cerca de 1.0 o menos,

al congelar la mezcla dentro de un rango de temperatura entre -2°C a cerca de -8°C ; y donde el paso de los efectos de la conversión a gel durante el congelamiento del almidón y

donde la confitería congelada tiene una mejora de derretimiento sobre la confitería congelada que no contiene el almidón como ingrediente.

17. El proceso de acuerdo con la reivindicación 16 donde el almidón es un almidón amilopectina.

18. El proceso de acuerdo con la reivindicación 17 el almidón amilopectina es un maíz ceroso.

19. El proceso de acuerdo con la reivindicación 16, que además comprende el paso de pasteurización de la mezcla dentro de un rango de temperatura de cerca de 70°C a cerca de 100°C .

20. El proceso de acuerdo con la reivindicación 16, que además comprende la homogenización de la mezcla dentro de un rango de presión de cerca de 2Mpa a cerca de 20Mpa.

21. El proceso de acuerdo con la reivindicación 16, que además comprende

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DEL MIN. INTERIOR Y DE JUSTICIA

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el paso de enfriar la mezcla dentro de un rango de temperatura de cerca de 2°C a cerca de 8°C.

22. El proceso de acuerdo con la reivindicación 16, que además comprende el paso de añejamiento de la mezcla durante un periodo de tiempo de cerca de 4 horas a cerca de 24 horas.

23. El proceso de acuerdo con la reivindicación 16, que además comprende el paso de endurecimiento de la mezcla a una temperatura de cerca de -20°C a cerca de -40°C.

24. El proceso de acuerdo con la reivindicación 16, que además comprende el paso de reemplazar por lo menos una parte de ingredientes sólidos de la confitería congelada con el almidón.

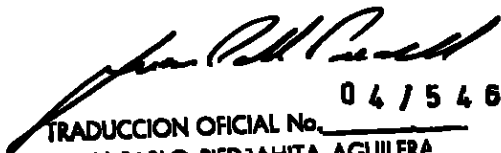
25. Un almidón funcional para uso en postres congelados, el almidón suministrándole al almidón del postre congelado una δ "tar" de 1.00 o menor, cuando se le formula en el postre congelado,

donde el almidón funcional es capaz de convertirse en gel durante condiciones de congelamiento, y

donde el almidón funcional suministra un derretimiento mejorado, frigidez reducida y/o expansión/contracción reducida del postre congelado, comparadas con un postre congelado preparado sin el almidón funcional.

ES TRADUCCIÓN FIEL Y COMPLETA de la cual se deja copia en los archivos de esta oficina para su confrontación y a la cual me remito.

Bogotá, Noviembre 29, 2004.


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TRADUCTOR E INTERPRETE OFICIAL
RES. 718 DE MAYO 2003
DEL MIN. INTERIOR Y DE J

2° EXAMEN DE FORMA, PATENTE DE INVENCION

Radicación N° **04-84717**

Folio ~~194~~

Concepto Técnico N° **701**

Clasificación Internacional de Patentes: **A23L 1/195**

Practicado el examen de acuerdo a lo establecido en el Art. 38 de la Decisión 486, se determinó que esta solicitud de **Patente**:

- SI ☒ NO ☐ Contiene un resumen de la invención (Art. 26-e)
- SI ☒ NO ☐ Contiene el título o nombre de la invención (Art. 27-d)
- SI ☒ NO ☐ Contiene la descripción de la invención (Art. 26-b)
- SI ☒ NO ☐ NO ES APLICABLE ☐ Contiene los dibujos necesarios para comprender la invención (Art. 26-d)
- SI ☒ NO ☐ Contiene una o más reivindicaciones (Art. 26-c)
- SI ☐ NO ☐ NO ES APLICABLE ☒ Han sido desarrollados los productos a partir de recursos genéticos o de sus productos derivados de los que cualquiera de los Países Miembros es país de origen (Art. 26-h)
- SI ☐ NO ☐ NO ES APLICABLE ☒ Los productos y o procedimientos han sido obtenidos o desarrollados a partir de los conocimientos tradicionales de las comunidades indígenas, afro americanas, o locales de los Países Miembros, de acuerdo a lo establecido en la Decisión 391 y sus modificaciones y reglamentaciones vigentes (Art. 26-i)
- SI ☐ NO ☒ La invención se refiere a un producto relativo a un material biológico (Art. 26-j)
- SI ☒ NO ☐ NO ES APLICABLE ☐ La prioridad coincide con la materia reivindicada.
- SI ☒ NO ☐ **CUMPLE LOS REQUISITOS TÉCNICOS**

OBSERVACIONES Y OTROS: SI ☐ NO ☐ Ver hoja(s) anexa(s)

EXAMINADOR TÉCNICO: 202051

Bogotá D C, septiembre 5 de 2005.

2020
Bogotá D C

Doctor(a):
DILIA MARIA RODRIGUEZ D'ALEMAN.
Apoderado (a)

REFERENCIA:	Radicación N°	04-84717
	Trámite	002
	Evento	001
	Actuación	416
	Oficio N°	856
	Folios	001

Teniendo en cuenta que la solicitud de privilegio de patente que se tramita bajo el expediente indicado en la referencia, reúne los requisitos de forma establecidos en los artículos 26 y 27 de la Decisión 486, de la Comisión de la Comunidad Andina, y en las demás disposiciones vigentes sobre la materia, se envía el extracto de la solicitud a la oficina de comunicaciones para efecto de su publicación en la Gaceta de Propiedad Industrial, conforme lo establece el artículo 40 de la Decisión 486.

Cúmplase
Dado en Bogotá D C,

12 SET. 2020


ALIX CARMENZA CÉSPEDES DE VERGEL
Jefe División de Nuevas Creaciones.

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