Cl. 99

Published May 4, 1943

Serial No. 296,157

ALIEN PROPERTY CUSTODIAN

EDIBLE ICE, AND THE PRODUCTION THEREOF

Anders Herlow, Grindsted, Denmark; vested in the Alien Property Custodian

No Drawing. Application filed September 22, 1939

This invention relates to edible ice, which means in the following specification and claims, products such as ice cream, milk ice and sherbet taken for refreshment as well as—in some cases and for certain kinds of ice—for nourishing purposes. I wish it to be understood that the invention comprises all kinds of such products irrespective of the term or name connected thereto. To make this understood I use the term edible ice, which comprises such kinds of refreshment and nourishing ices whereas it is not intended to cover thereby the pure frozen water, cristal ice socalled. The term edible ice comprises soft ices as well as the hardened ices sold for instance in pails, boxes or the like containers or in cartons, packages, wafers, etc.

It is well known in the production of edible ice to add to the mass to be frozen stabilisers such as gelatine, agar-agar, pectin, and the like as well as yolk of egg in the fresh or desiccated state as well as milk powder or condensed milk. It has been found that particularly the two latter additions, viz. dry substance of egg-yolk and dry substance of milk will contribute to make the flavour of the finished ice rich and to obtain an improved expansion of the mass.

The improvement obtained by the addition of dry substance of egg yolk or milk, in view of the facts which will be explained in the following, must be assumed to be due to some extent to the face that yolk of egg and milk powder contain phosphatides which are surface active substances containing in their molecule groups that are lipophillic and aerophillic as well as groups that are hydrophillic.

By adding egg yolk or milk powder to the mass there is incorporated therein not only the surface active phosphatides but also other substances such as fats, proteins, sugars, salts and flavouring substances which in some cases is undesirable. Moreover the employment of yolk of egg or milk powder is rather expensive when nothing but the surface activity of these substances is made use of. Thus there must normally be used one egg yolk or 5 grms. dried yolk of egg for 1 liter of the mass to be frozen in order to obtain a substantial effect.

The object of the present invention is on the first hand the production of edible ices in which the taste is richer and more fatty and the texture is finer than in known ices, the expansion being at the same time as great or greater as in known ices the flavour of which is not so rich and the texture of which is not so fine and smooth.

A more special object of my invention is the production of an edible ice comprising a fat emulsion, the fat particles forming the dispersed phase thereof, sugar, a hydrophillic colloidal substance acting as stabiliser and flavouring substances, such edible ice having the properties mentioned above.

Another more special object of my invention is the production of an edible ice comprising sugar, a hydrophillic colloidal substance acting as a stabiliser and flavouring substances together with water, such edible ice having the properties mentioned above.

Further objects of my invention will appear from the following description.

With such objects and purposes in view the difference between the edible ices produced according to my invention and known ices and the difference between known methods of producing such ices and the methods according to my invention resides mainly in the fact that substances different from phosphatides and containing in their molecule groups that are lipophillic and aerophillic and groups that are hydrophillic are added to the mass to be frozen. Such substances in view of the presence of the said groups in their molecules are surface active or interphase active and act as dispersing agents for which purpose they are employed in many industries.

It has been found that by adding such dispersing agents which must be edible in the concentration in which they are to be employed the texture of the ice will be finer and more smooth and the flavour will be more rich and fatty. This will be the case even when the ice be expanded more than has hitherto been normal practice. The distribution of the air and of the ice cristals as well as that of the fat particles in the cases where fat is present will be finer. The use of yolk of egg may be dispensed with, if so desired, and the results that can be attained will be better than those obtained with egg yolk. If milk powder or condensed milk is otherwise added for dispersing purposes only, the addition thereof can also be dispensed with.

The mass from which edible ices are produced by freezing are normally emulsions of the type oil-in-water. The dispersed phase in many cases for instance in the case of cream ice, consists mainly or exclusively of the fat balls of the cream. Even in the case of other types of ices, for instance in the case of milk ice, emulsified fat may be present in the mass. On the other hand types of edible ices are known which are produced by freezing a mass which is not an emulsion of fat. Thus edible ice may be produced by freezing an aqueous solution of for instance fruit juice and sugar. In the mass of such edible ices as well as of that produced from emulsions of fat, hydrophillic colloids are normally present, for instance pectin substances, agar-agar or gelatine. By the expansion step of the production, however, air is incorporated in the mass which in some cases is also brought to contain carbon dioxide in the dispersed phase.

The product subjected to freezing and in some cases to hardening will accordingly in all cases be in the nature of a disperse system of the type oil-in-water or air-in-water or normally both oil-in-water and air-in-water.

For the dispersing agent such substances are accordingly preferably used which have in themserves the tendency of producing emulsions of the type oil-in-water and such dispersing agents will normally also be dispersing agents for air, the lipophilic group of such substances being at the same time aerophilic.

It is well known, however, that many dispersing agents can be successfully used in the production of emulsions of the type oil-in-water as well as in the production of emulsions of the type water-in-oil. Accordingly nothing will prevent employing in accordance with the present invention such dispersing agents as well as dispersing agents which are normally particularly suited for use in the production of emulsions of the type water-in-oil, provided that care is taken to see that the dispersion obtained is of the type oil-in-water and air-in-water. For this purpose the dispersing agent may be added to the mass to be frozen previous to or during freezing in the shape of a pre-emulsion or dispersion of the oil-in-water or air-in-water type. In the production of this pre-emulsion care is taken that the ratio between the proportions of fat and water is so as to be favourable for the formation of a dispersion of the desired nature and, if necessary, the production of such a dispersion is secured in other manners known per se, for instance by suitable mechanical and physical influencing.

In many cases, for instance in the case when monoglycerides are used for the dispersing agent it has been found that the melting point is of importance, the results obtained being the better the higher the melting point within reasonable limits.

The explanation of this fact is presumably that that the melting point of the dispersing agent shall preferably be higher than or in the vicinity of the temperature at which the mass or the pre emulsion is subjected to the substantial part of the treatment by which the dispersion is obtained or brought into the desired degree of dispersity which treatment is normally a process of the kind called homogenizing. The emulsion in this case will be prevented from forming or returning to a water-in-oil emulsion by the complete or partial gelation of the films produced round the balls of fat. The said selection of the melting point of the dispersing agent may therefore be considered a precaution to secure the formation of dispersions of the type oil-in-water.

In some cases this purpose can also be attained by employing a lower homogenisation temperature than usually.

In the following the employment of a number of different dispersing agents is described in connection with the production of edible ices of three different types, the manner in which the mass is prepared and the manner of expanding and freezing it as well as the kind and proportions of the ingredients of the mass being similar in the case of a great number of the dispersing agents mentioned in order to render possible a comparison of the effect of the same. It goes without saying that the invention is not limited to the specific operations described or the order of succession thereof or the specific ingredients or proportions mentioned.

An evident variation of the method described is the employment of ice cream powder by which cream, milk or water has added to it the other constituents of the mass to be frozen in the shape of a powder. This powder will thus contain for instance dry substance of milk together with sugar, gelatine, algine or other protective colloids and flavouring substances and carbon-dioxide-producing constituents, if so desired. Evidently, according to the present invention, the dispersing agent may be present in the ice cream powder, but preferably such dispersing agents are to be used which tend to produce oil-in-water emulsion, i. e. especially the dispersing agents mentioned in Example I.

The precautions mentioned in the example and serving to improve the fineness of the emulsion, viz. the homogenisation, are not indispensable, the use of a dispersing agent causing the dispersion to be finer, even when these precautions are not made use of.

Example I

A cream mixture corresponding to what has been termed mass in the preceding part of the specification is produced from:

	Kgs.
Cream	200
Sugar	14
Condensed milk	24
Gelatin	1,5

Colour and flavouring substances, suitable quantities.

Dispersing agent as indicated below.

The mixture is passed through a homogeniser at 60–70° C. and the homogenised mixture is left to stand for maturing at low temperature, preferably 2–4° for 24 hours. In place of the homogeniser other means for improving the emulsion with respect to its stability and the size of the dispersed particles may be used.

After maturing the mass is introduced into an ice-cream freezer which is a container surrounded by a cooling jacket and mounted with a rotating whipping and scraping member. In this freezer a certain portion of the mass at a time is treated for a period of 6–20 minutes with a cooling medium of —20 to —35° C. being passed through the jacket and the treatment is discontinued when the desired consistency and temperature has been attained. The portion of the mass introduced in the container at the commencement of the treatment will fill the same partially only but during the treatment the volume will increase owing to the expansion or swelling, so-called.

After the treatment in the freezer the mass is introduced into pails or moulds which are frozen and hardened for 24 hours at 120 to —25° C. except in the case of soft ice when this freezing and hardening is dispensed with.

For the purpose of comparison five masses in which the contents of dispersing agent are as follows are frozen in the manner described above.

	Per cent
(*a*) No dispersing agent	
(*b*) Dried yolk of egg	0,5
(*c*) Sodiumcetylsulphate	0,1
(*d*) Monostearic acid triglycerin	0,2
(*e*) Mixture of 50% monostearic acid glycerin and 50% monostearic acid diglycerin	0,3

During the treatment in the freezer the same expansion was aimed at in all five cases, viz.

100% but this degree of expansion was obtained only with difficulty in case (*a*) when no dispersing agent was added. In the other four cases (*b*)–(*e*) it was easily obtained.

The quality of the product obtained was decidedly better when egg yolk was used than without egg yolk. In the three experiments (c)–(*e*) in which a synthetic dispersion agent has been used, the quality was still appreciably better than in the case of yolk of egg. The consistency may be characterised in the following manner.

(a) Watery and rather spongy.
(b) Smoother and more rich in flavour.
(c) Smooth, velvety and of a more fat taste.
(d) Similar to (c).
(e) Smooth, velvety, very rich and fat in flavour.

The improvement is partially due to the fact than can be found at microscopical analysis that the dispersion of the bubbles of air is finer and that the fat is present in more balls of smaller size.

Generally the result will be further improved when the dispersing agent is homogenised together with only a small part of the mass which is then added to the balance of the mass when the same has been homogenised.

Example II

A dispersing agent is added to a mass of the same composition as in Example I, in the following manner.

The dispersing agent is added in the molten state at 70–80° C. or in the shape of a powder or a paste formed with water and in the proportion stated below to 5 litres of the mass at 65° C. After the dispersing agent has been preliminary dispersed by whipping or agitation the mixture is passed for 10 minutes through a homogeniser at 200 atm. when it will have passed several times through the pressure head of the homogeniser.

After homogenisation the pre-emulsion is cooled to 20° C. and added to the balance of the mass which has in the meantime been homogenised. This homogenisation may be dispensed with, if so desired. As dispersing agent one of the substances or mixtures stated below is added in the proportion stated.

	Per cent
(*a*) Monopalmitic acid diglycerol	0,2
(*b*) Monopalmitic acid glycerol	0,4
(c) Mixture of 9 parts monostearic acid diglycerol and 1 part sodium salt of monosulphoacetic acid monopalmitic acid glycerol with 10 parts of water to form a paste	0,5
(*d*) Monostearic acid polysaccharide from dextrine	0,2
(*e*) Monostearic acid saccarose	0,2
(*f*) Monostearic acid glycose	0,2
(*g*) Mixture of 9 parts monostearic acid glycerol and 1 part stearate of lys-albic acid with 10 parts of water to form a paste	0,5
(*h*) Pentahydroxyethylether of monostearic acid sorbitol	0,2
(*i*) Sodium salt of monopalmitic acid monophosphoric acid glycerol	0.2
(*k*) Monoglyceride of slightly oxidised, partially hardened fatty acid of soy oil	0,2
(*l*) Monoglyceride of hardened fatty acid of castor oil	0,2
(*m*) Stearic acid aminoacetic acid monoglyceride	0,2

From the mixture a cream ice is produced in the manner described under Example I, the result being in all cases that the product is of finer texture and richer and more fatty in taste than when no dispersing agent of the kind described is used. Moreover the expansion of 100 per cent aimed at is obtained with facility, contrary to the mixture when no dispersing agent is present of the kind described. Even a degree of expansion of 110–120% is readily obtained without the texture and flavour being influenced.

Example III

The mass is of the same composition as stated in Example I, except for the cream which is replaced by the same proportion of milk.

For the dispersing agent are employed for instance the following substances which are added in the manner stated in Example II:

	Per cent
(*a*) Monostearic acid glycerol	0,5
(*b*) Monostearic acid diglycerol	0,3

After expansion freezing and hardening in the manner stated in Example I a milk ice is obtained. The difference between such milk ice prepared without dispersing agents and with dispersing agent of the kind stated is even more appreciable than in the case of cream ice.

Example IV

For the production of water ice (sherbet) a mass of for instance the following constituents is prepared.

	Kgs.
Juice of raspberry (whole raspberries crushed with 50% sugar)	15
Sugar	8
Water to dissolve sugar	4
Water	26
Gelatin	0,2
Water to dissolve gelatin	2

In accordance with the invention a dispersing agent is added, for instance 250 grms. monostearic acid glycerol mixed with 2 kgs water.

The mass is frozen as stated in Example I with the exception that the expansion is only about 50%, so that 30 liters of ice are obtained from 20 liters of the mass. Without dispersing agent only 25% expansion can be obtained with the same mass i. e. 25 liters of ice from 20 liters mass. The ice in which a dispersing agent is present, is moreover of a finer crystalline texture, and when the ice is moulded with a stick in it, it will fasten better to the stick.

In the types of edible ice given in Examples I–IV various flavouring substances may be substituted for the ones mentioned. Moreover, the invention may readily be employed with other recipes, thereby obtaining similarly favourable results. Thus the invention may be employed in connection with the types of ice frozen in the presence of dissolved and dispersed carbon dioxide, irrespectively whether it is added in the freezer or already present in the mass.

In each of the examples given the dispersing agents mentioned in the other examples may be used with the exception that not all of the dispersing agents mentioned in Example II can be added directly as mentioned in the first part of Example I.

Similarly other dispersing agents may be used in the examples described as well as with other recipes or types of ices, thereby obtaining similar results.

Synthetically produced dispersing agents, fulfilling the conditions of being used according to the present invention in that they contain at least one hydrophillic group and at least one lipophillic group and in that they are not poisonous and taste-spoiling in the concentration in which they are to be used, are to be found in very great number on the market and a still greater number is described in literature. Such substances are used to a great extent in the production of dispersions of widely varying kind, for instance cosmetical or pharmaceutical emulsions, emulsions of fat to make substitution products to replace milk or cream or to make auxiliary means for bakery purposes in the production of margarine as detergents as wetting agents, in the textile industries or for other purposes, for the production of foam etc.

It is not possible giving a compendiary description of all possibilities of composing such substances, and no grouping can be, so far, be given comprising all such substances. Accordingly the following statements when they are general in form as well as when particular chemical individuals are mentioned shall not be taken for an exhaustive definition or enumeration of said substances but they are given for guidance only and are preferably chosen among the groups which are so far of the greater importance.

For the lipophile groups there may be used higher fatty acid or higher fat alcohols or the derivatives thereof soluble in fat or, somewhat more generally, hydrocarbon chains or rings soluble in fat.

Examples of hydrophillic groups that may be used in the composition of the dispersion agent are acid groups, which may be neutralised or not, hydroxyl groups and amino groups. Thus the following group are very active: sulphuric acid groups or phosphoric acid groups or free hydroxyl groups in divalent or polyvalent alcohols or the derivatives thereof or in sugars, such as monosaccharides, disaccharides, trisaccharides and polysaccharides or hydroxy alkyl ethers, in which free hydroxyl groups are present in the alkyl etherified with the alcohol. Even the hydroxyl group of higher monovalent alcohols, for instance cetyl alcohol, are effective to some extent. In this latter compound the hydrophillic group is bound directly to the lipophillic group but the hydrophillic and lipophillic groups may be connected in many other ways among which condensation, etherification, esterification, connection through an oxygen bridge and connection through nitrogen-containing groups as for instance in amino acids may be mentioned. Also oxygen groups and peroxide groups are hydrophillic.

In lipophillic groups connected to hydrophillic groups other hydrophillic groups may be introduced, for instance the oxygen group produced in an unsaturated fatty acid chain by blowing the substance in which it is contained at a suitable high temperature with an oxygen-containing gas. Other examples are sulphonic acid, sulphuric acid, phosphonic acid or phosphoric acid groups that may be neutralised or not. Moreover the fatty acid chain of an oxy acid may be used for the aerophillic and lipophillic group.

In the examples monopalmitic acid and monostearic acid glycerol are mentioned as excellent dispersing agents for the present purpose. The same applies to other monofatty acids glycerols in the case of higher fatty acids. It is also mentioned that monofatty acid diclycerol and triglycerol are effective and this applies also to monofatty acid polyglycerol.

In the production of these substances alkali salts, particularly sodium or potassium salts of fatty acids are commonly employed for catalytic purposes, even salts of the lower fatty acids, for instance sodium acetate. Accordingly such salts will frequently be present in small proportions in the dispersing agents in question, for instance in a proportion of abt. 2% of the dispersing agent. The alkali salts of the higher fatty acids, for instance sodium stearate, are moreover dispersing agents which in themselves may be used according to the present invention, provided that the proportion thereof is so small that it does not spoil the taste of the product. Just as glycerol in which one or two hydroxyl groups are left free glycol in which one hydroxy group is left free and the derivatives thereof for instance di-, tri- and polyglycol may advantageously be used for the hydrophillic group of the dispersing agent. Excellently suited is for instance monofatty acid, triethylene glycol. When a hydroxy compound having more than two alcohol groups of which at least one is left free is used, the other alcohol groups may be esterified with the same or different lipophillic acid groups.

Among the groups that may be used in the lipophillic part of the molecule of the dispersing agent sterols, such as cholesterol, may be mentioned as well as hydro carbons which are derivatives thereof and soluble in fat.

When lysalbic acid is mentioned among the examples it must be understood that amino acids and the decomposition products of proteins are generally strongly hydrophillic and they may, therefore, be used in the hydrophillic part of the molecule of dispersing agents for the present purpose.

The finer distribution of fat, if present, and of air causes the walls of the air particles to be thinner. In some cases, therefore, it will be necessary to strengthen them, which can be done by adding a stabiliser or increasing the proportion of stabiliser which would otherwise have been used. The dispersing agent may also be used in mixture with a stabiliser such as gelatine, algine, agar-agar, pectine etc. in a proportion suitable to compensate for the deficiency in stabiliser produced in the addition of the dispersing agent, as explained above. Example: equal parts of dispersing agent and stabiliser to 2 parts of stabiliser to 1 part if dispersing agent.

ANDERS HERLOW.