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(54) Title: METHOD FOR MELTING FROZEN, WATER-CONTAINING PRODUCTS

(57) **Abrégé/Abstract:**

The invention relates to a method of thawing frozen, water-containing products, in which frozen product is heated and at the same time stirred in a horizontal mixer.



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Method for melting frozen, water-containing products

The invention relates to a method of melting frozen, water-containing products, in particular protein-containing products, using a mixer having horizontal internals.

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The method is suitable for the gentle melting of water-based ice which contains, in particular, both mechanically and thermally sensitive substances.

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Especially biological products which can only be produced in small quantities have to be temporarily stored before further processing. This is usually carried out by means of freezing in order to maintain the quality of the product during storage. The controlled thawing of the products serves not least to even out the product properties which, particularly in the case of biological products, can vary greatly from batch to batch. To achieve a small range of fluctuation of the product

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properties, it is frequently necessary for several hundred to a few thousand batches to be thawed together and mixed with one another.

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This is frequently carried out in vertical stirred vessels with jacket heating, because in this way no seal is touched by the product when the stirrer is operated from above. The ice pieces are taken from the storage containers and introduced into the stirred vessel. Breaking up the ice pieces is often not advisable for hygienic reasons. It is frequently also the case that no additives are required or are even permitted, so that the melting process in the stirred vessel takes place initially without a liquid phase. The ice pieces are set into rotary motion by the stirrer and

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slide along the heated wall of the vessel. The molten material collects at the bottom of the vessel. Owing to the size of the ice pieces relative to the internal diameter of the stirred vessel (the ratio of edge length to diameter can even exceed 1/10), the installation of baffles is not advisable. There is therefore a risk of the ice pieces jamming and leading to mechanical failure of the stirrer.

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For this reason and to intensify heat transfer via the wall, stirrers which travel around the wall, for example helical stirrers, are usually employed. Owing to the low friction between water ice and the wall of the vessel due to formation of a water film and the movement of the stirrer used around the wall, the ice pieces are

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not mixed with one another. In extreme cases, it is even possible for ice pieces which have previously been partially thawed on the external surfaces but are still deep frozen on the inside to freeze together and form a coherent mass which rotates together with the stirrer without any relative motion. The ice pieces which touch the wall melt at the wall until an air gap having a width of a few millimetres

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has been formed. In this mode of operation, there is hardly any formation of fresh contact areas, so that the thawing process takes longer. During this process, the thawed material is subjected to a high thermal stress for a prolonged time, which can have an undesirable effect on the product properties.

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In all cases, the ice pieces float on the surface because of their lower density compared to the molten material. The previously mentioned low wall friction leads to the impetus necessary for axial transport not being able to be introduced. A division into a molten fraction in the lower region and still solid ice pieces rotating together with the stirrer in the upper region is the result. Heat exchange between these two regions is hindered by the lack of axial transport of the ice pieces, which has the consequence that the molten product is subjected to a high thermal stress over a long period of time.

10 The invention accelerates the melting process of mechanically and thermally unstable substances in aqueous solutions and to avoid mechanical and thermal stress as far as possible. Particular attention was to be paid to ensuring that local overheating due to separation of solid and liquid phases is nevertheless avoided at a low specific power input as high space-time yield by means of low stirrer speeds.

15 This is achieved by a method which prevents separation of the two phases by use of a horizontal mixer in which the stirrer elements do not rotate about a vertical axis but make vertical mixing possible.

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The invention provides a method of thawing frozen, water-containing products, in particular protein-containing products, by introducing the frozen product into a horizontal mixer, heating the mixer and at the same time regulating the contents of the mixer intensively, with any floating frozen product being continually submerged in the liquefied phase and mixed with this.

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Preference is given to a method in which the mixing by heating is continued until the product has been melted completely.

35 Preference is also given to a method in which frozen product is continuously introduced into the horizontal mixer, mixing and heating are carried out continuously and a thawed product is taken off in the same amount so that a steady state is established. Such a continuous method has the advantage that control of the

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temperature is simplified and the residence time is shortened and the thermal stress on the product is thus reduced.

5 For the purposes of the invention, a horizontal mixer is a dynamic mixing apparatus which has a stirrer (movable internals) and whose axis or axes (in the case of a plurality of stirrer shafts) of rotation are arranged essentially horizontally. This will be taken to encompass an apparatus whose axis of rotation of the stirrer shafts is at an angle of at least 45° to the vertical.

10 Preference is given to a method in which a disc mixer, ploughshare mixer or Becker mixer is used as horizontal mixer.

Further preference is given to a method in which mixing elements having internal heating are used as horizontal mixer.

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Particular preference is given to using a horizontal mixer which has wiping elements travelling around the wall, in particular a mixer having a self-cleaning factor of at least 80%, in the method.

20 Further preference is given to a method in which protein-containing solutions and suspensions from natural biological sources or from biological processes, particularly preferably blood plasma, are used as water-containing product.

25 In the method of thawing blood plasma or blood plasma products, the temperature of the mixture is maintained at less than 10°C above the melting point, preferably less than 5°C above the melting point, during the entire process.

The method can be carried out either batchwise or continuously.

30 In continuous operation, completely melted product is taken off from the mixer and further frozen product is introduced batchwise.

The continuous method reduces the residence time of the products in the melting zone and is particularly useful for the treatment of particularly sensitive products.

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In the method of the invention, floating ice pieces are conveyed upwards from the surface of the melt and are randomly conveyed by gravity to other places in the mixer. Secondly, they are pushed downwards into the molten phase which is in the

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process of warming. The forced vertical relative motion of the frozen products significantly improves the heat transfer between the frozen phase and the liquid phase. This also leads to higher temperature gradients at the wall and thus to an overall shorter melting time compared to the methods known from the prior art.

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ExamplesExample 1

5 Water ice in cylindrical form having volumes of about 20 ml was introduced into a cylindrical vessel having a diameter of 200 mm and jacket heating. The degree of fill was varied in the range from 40 to 100%. Here, a degree of fill of 100% corresponded to the maximum charge of water ice in the frozen state. The vessel was firstly operated vertically, then horizontally. The speed of rotation of the
10 mixer was maintained at a constant 30 min^{-1} in all experiments. The jacket heating was set to a water temperature of 20 or 30°C. The thawing process was observed by means of a temperature sensor in the cylindrical vessel. In vertical operation, there was insufficient vertical exchange between liquid and ice, resulting in long thawing times. The relevant thawing time was determined by attainment of a
15 particular temperature, measured by the temperature sensor in the cylindrical vessel. In the case of horizontal operation, good mixing between previously thawed water and still frozen ice was achieved by the vertical movement of the mixing blades. The heating area available was always greater in vertical operation than in horizontal operation. At a degree of fill of 40%, the measured thawing
20 times were able to be halved. At the maximum degree of fill, the thawing times were reduced by a factor of < 0.8 . The significant reduction in the thawing time is a consequence of the better mixing in horizontal operation, because the internal heat transfer coefficient at a constant temperature difference is increased as a result and the reduction in the heat transfer area can be more than compensated for.

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Example 2

30 In a second experiment, frozen human blood plasma in pieces of about 50 ml was introduced into the same experimental vessel and thawed under otherwise identical conditions. The temperature curves at degrees of fill of 70% displayed the same significant effect of the halving of the thawing times. Apart from the temperature curves, the activity of a protein (factor VIII) which precipitates during the thawing
35 process was analyzed. As a result of the shorter thawing times and the lower thermal stress due to the more homogeneous temperature field in the cylindrical vessel, activity increases of generally 20% or more were obtained in horizontal operation compared to vertical operation.

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CLAIMS:

1. A method of thawing a frozen, water-containing product, comprising introducing the frozen, water-containing product into a horizontal mixer, heating the mixer and at the same time mixing any thawed products intensively, with any floating
5 frozen, water containing product being continually submerged in a liquefied phase and mixed with the liquefied phase.
2. The method according to claim 1, wherein the frozen, water-containing product comprises proteins.
3. The method according to claims 1 or 2, wherein a disc mixer,
10 ploughshare mixer, or Becker mixer comprises the horizontal mixer.
4. The method according to any one of claims 1 to 3, wherein mixing elements comprising internal heating are used.
5. The method according to any one of claims 1 to 4, wherein the horizontal mixer comprises wiping elements travelling around the wall.
- 15 6. The method according to any one of claims 1 to 5, wherein the frozen, water-containing product comprises a protein-containing product from a natural biological source or from a biological process.
7. The method according to claim 5, wherein the temperature of the frozen, water-containing product is maintained at less than 10 °C above the melting
20 point of the frozen, water-containing product during mixing.
8. The method according to any one of claims 1 to 5, wherein the horizontal mixer is operated continuously.
9. The method according to claim 7, wherein the temperature of the frozen, water-containing product is maintained at less than 5 °C above the melting
25 point of the product during mixing.