

Claims

1. A method of drying a frozen product stored in a container (703) having a container wall defining a cavity for holding said product, the method being a method of drying by sublimation or being a method of drying by desorption, the method comprising the steps of:
 - 5 a) capturing (1401) a thermal IR image of at least a portion of the container wall using at least one thermal IR camera;
 - b) processing (1402) the thermal IR image by determining a plurality of temperature values associated with a plurality of points located on an outer surface of the container wall, using an image processing module (702);
 - 10 c) calculating (1403) a maximum temperature of the product (T_{prod_max}) in the container using a mathematical model that models heat flow and that models progress of the drying process;
 - d) controlling an amount of power supplied to at least a portion of the container based on the calculated maximum product temperature (T_{prod_max}) and on a temperature safety_margin (T_{sm});
 - e) repeating at least once the steps a) to e).
- 15 2. The method according to claim 1, wherein the method further comprises:
 - step f) preceding step d) of determining (1404) a temperature safety margin (T_{sm}) as a temperature difference between the temperature of the product (T_{prod}) and a predefined critical temperature (T_{crit}) related to the product based on said calculated progress.
3. A method according to claim 2, wherein step f) comprises calculating the temperature safety
 - 20 margin (T_{sm}) using the mathematical model by taking into account at least one of:
 - a predetermined content of said product;
 - at least a subset of the temperature values calculated in step b);
 - an estimated or calculated cumulative amount of heat energy provided to or absorbed by the container.
- 25 4. A method according to any of the previous claims,
 - wherein the container has a longitudinal axis and is rotated around its longitudinal axis and has a substantially circular cross-section in a plane perpendicular to said longitudinal axis;
 - and wherein the mathematical model is mainly based on heat transfer from an outside of the container wall, through the container wall, and through a portion (Z1) of the product still containing ice
 - 30 crystals.
5. A method according to any of the previous claims, wherein the method of drying is a method of sublimation, and wherein the mathematical model is based on one of the following models:
 - A) a model of supplying heat energy to a body comprising three concentric cylindrical shapes, comprising:
 - 35 a) an outer cylinder (105) formed by the container material;

- b) an intermediate cylinder (101) in physical contact with the outer cylinder (105) and comprising frozen product still containing ice crystals;
 - c) an inner cylinder (103) containing frozen product substantially free of ice crystals;
 - or
 - 5 B) a model based on supplying heat energy to a body comprising a plurality (N) of at least two disks (176, 177), each disk comprising three concentric annular rings (175, 171, 173) comprising:
 - a) an outer ring (175) formed by the container material;
 - b) an intermediate ring (171) in physical contact with the outer ring (175) and comprising frozen product still containing ice crystals;
 - 10 c) an inner ring (173) containing frozen product substantially free of ice crystals.
- 6. A method according to any of the claims 1 to 5, wherein the method of drying is or further comprises a method of desorption, and wherein the mathematical model is based on one of the following models:
 - A) a model of supplying heat energy to a body comprising three concentric cylindrical shapes,
 - 15 comprising:
 - a) an outer cylinder (105) formed by the container material;
 - b) an intermediate cylinder (101) in physical contact with the outer cylinder, comprising product substantially free of ice crystals, and substantially free of moisture content;
 - 20 c) an inner cylinder (103) containing product substantially free of ice crystals but still containing moisture content;
 - or
 - B) a model of supplying heat energy to a body comprising a plurality (N) of at least two disks (176, 177), each disk comprising three concentric annular rings:
 - 25 a) an outer ring (175) formed by the container material;
 - b) an intermediate ring (171) in physical contact with the outer ring (175) and comprising product substantially free of ice crystals, and substantially free of moisture content;
 - 30 c) an inner ring (173) containing product substantially free of ice crystals but still containing moisture content.
 - 7. A method according to any of the previous claims, wherein the container has a side wall portion having a cylindrical shape or a conical shape or a truncated conical shape or a paraboloid shape or a truncated paraboloid shape (221) over at least a portion of the height of the container.
 - 8. A method according to any of the previous claims, wherein step d) comprises one or more of the following actions:

- i) controlling an amount of power supplied to the at least one heater (705);
 - ii) controlling a distance (dh) between the at least one heater (705) and the cylinder (703);
 - iii) controlling and orientation between the at least one heater and the cylinder;
 - iv) controlling an exposure time of the container in front of the at least one heater;
 - 5 v) controlling the translational and/or rotational movement of the cylinder.
9. A method according to any of the previous claims,
- wherein step d) comprises controlling the amount of power supplied to the container by controlling at least a first amount of power provided to a first heater (1803) and by controlling at least a second amount of power provided to a second heater (1804), located at a different position relative to
- 10 the container.
10. A method according to any of the previous claims, wherein the at least one heater (FIG 8: 705) is movable relative to the container (703).
11. A method according to any of the previous claims,
- wherein step d) comprises estimating or calculating at least one temperature (Tprod) of at least
- 15 one point of the product located in the intermediate cylinder (101) or in the intermediate ring (171) using the mathematical model; and
- wherein controlling the at least one heater (703) comprises controlling said heater such that the product temperature (Tprod) is smaller than or equal to a critical temperature (Tcrit) minus the safety margin (Tsm).
- 20 12. A method of freeze-drying a liquid product, comprising:
- g) providing a container;
 - h) inserting the liquid products in said container;
 - k) freezing the product in said container while rotating the container about its longitudinal axis at a predefined speed;
- 25 - applying a first drying step for removing ice crystals from the product, using a method according to any of the claims 1 to 11, or applying a second drying step for removing moisture content from the product, using a method according to any of the claims 1 to 11, or both.
13. A method of freeze-drying a liquid product according to claim 12,
- wherein step g) comprises providing a container containing a side wall portion (221) having a
- 30 substantially constant thickness and having a substantially paraboloid shape or a truncated paraboloid shape over at least a quarter of the height of the container; and
- wherein step k) comprises freezing the product in said container while rotating the container about its longitudinal axis at a predefined speed chosen corresponding to the curvature of the paraboloid shape, such that the product will form a layer of substantially constant thickness against the
- 35 side wall.

14. A freeze-drying apparatus (700) for drying a frozen product stored in a container (702) having a container wall defining a cavity holding said product, the apparatus (702) being adapted for drying said product by sublimation and/or by desorption, the apparatus comprising:

5 a) a thermal IR camera (701) for capturing a thermal IR image of at least a portion of the container wall;

b) an image processing module (702) adapted for processing the thermal IR image by calculating a plurality of temperature values associated with a plurality of points located on an outer surface of the container wall;

10 c) at least one heater (703, H1, H) arranged for heating at least a portion of the outer surface of the container wall;

- at least one of the following components: means for supplying power to the at least one heater (H1), means (715) for moving the at least one heater (H1), means (704) for moving the container (703);

d) a controller (713) adapted for repeatedly:

15 * calculating a temperature of the product (T_{prod}) in the container using a mathematical model that models heat flow and models progress of the drying process;

* calculating a temperature safety margin (T_{sm}); using a mathematical model that models that models heat flow and progress of the drying process of said product in said container;

* calculating a temperature safety margin (T_{sm}) between the temperature of the product (T_{prod}) and a predefined critical temperature (T_{crit}) related to the product;

20 * controlling an amount of power supplied to at least a portion of the container (702) by controlling at least one of the means for supplying power, the means (715) for moving the at least one heater, and the means (704) for moving the container (703).

15. A kit of parts comprising a freeze-drying apparatus (700) in accordance with claim 14 and a container (2200) suitable for use in said freeze-drying apparatus,

25 the container (2200) having a longitudinal axis, and comprising a container wall defining a cavity for holding a product to be freeze-dried;

the container wall having a bottom portion (2203) and at least a lower side portion (2201) and optionally an upper side portion (2202);

30 the lower side portion (2201) having a substantially constant thickness over at least a portion of its height;

a cross-section of the lower side portion (2201) in a plane containing the longitudinal axis defines at least one substantially parabolic shape or truncated parabolic shape;

a cross section of the lower side portion (2201) in a plane perpendicular to the longitudinal axis having a substantially circular shape.

16. A kit of parts in accordance with claim 15, wherein said container (2200) comprises a frozen pharmaceutical composition, or a frozen biological composition, or a frozen cosmetic composition or a frozen medical nutritional product located at an inner surface of said side portion.