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División de Nuevas Creaciones

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71. **SOLICITANTE** AGRESEARCH LIMITED

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74. **APODERADO** JUAN PABLO CADENA SARMIENTO

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☐ Capítulo I. ☒ Capítulo II.

2 SOLICITANTE (71)	Nombre: **AGRESEARCH LIMITED** Dirección: East Street, Ruakura Campus, Hamilton, Private Bag 3123, 2020 Hamilton, Nueva Zelanda Nacionalidad o Domicilio· Nueva Zelanda Lugar de Constitución· Nueva Zelanda Teléfono: Fax: E-mail	**IDENTIFICACION** C.C. ☐ NIT ☐ C.E. ☐ Otro ☐ Cual_____ Número ________
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10 **ANEXOS**

- [x] Comprobante de pago de la tasa de presentación de la solicitud. $443.000
- [ ] Documento que acredita la existencia y representación legal cuando el solicitante sea persona jurídica
- [ ] Poderes, si fuere el caso
- [ ] Documento de cesión del inventor al solicitante o a su causante.
- [ ] Declaraciones
- [x] Copia de la solicitud internacional. (Resumen, descripción, reivindicaciones, dibujos)
- [x] Traducción de la solicitud internacional al castellano. (Resu men, descripción, reivindicaciones, dibujos)
- [x] Copia del examen preliminar efectuado por la IPEA.
- [x] Traducción del examen preliminar efectuado por la IPEA
- [ ] De ser el caso, copia del contrato de acceso
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- [ ] De ser el caso, certificado de depósito del material biológico.
- [ ] Arte final 12x12.
- [ ] De ser el caso, información sobre otras solicitudes de patente o títulos obtenidos en el extranjero por el mismo titular o su causante, relacionadas parcial o totalmente con la invención de esta solicitud.
- [x] Otros - Documentos PCT
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11 **FIGURA CARACTERÍSTICA:**

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FIRMA: Juan Pablo Cadena

C.C. 80.410.337 de Usaquén T.P 57492 del C.S.J

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TRASPASO A

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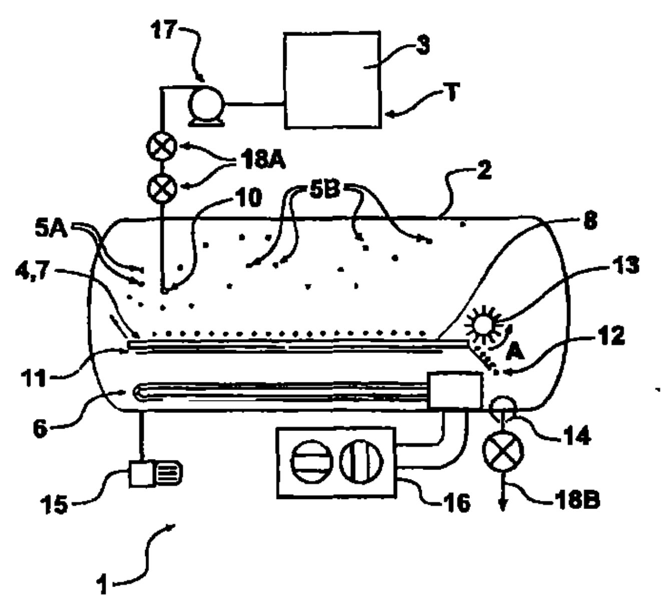
(54) Title: DRYING PROCESS AND APPARATUS



(57) Abstract: This invention relates to a process and/or method and associated apparatus for use in spray freeze drying a liquid substance such as fruit juice, pharmaceuticals, nutraceuticals, tea and coffee. A liquid substance is sprayed (atomised) and reduced in temperature to below its eutectic temperature; thereby inducing a phase change of liquids in the liquid substance. The atomised liquid substance (ALS) is then subjected to a vacuumous drying chamber in which the ALS is subjected to an energy source to assist sublimation of liquid from the ALS as it is conveyed through the chamber. Such an energy source can provide a temperature gradient through which the ALS passes and is exposed to, all carried out so as to preferably minimise or reduce the likelihood of degradation of the substance being dried (reducing the liquid content).

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

DRYING PROCESS AND APPARATUS

TECHNICAL FIELD

This invention relates to a dryer and a drying process. In particular, though not solely, this invention relates to a dryer for use in freezing, concentrating or drying a liquid substance having solid particles in suspension or a substance dissolved therein.

BACKGROUND ART

The process of food preservation has always been of interest, with freeze drying being of particular food industry focus since the late 1940's. In the formative years of freeze drying apparatus and processing development, there was an emphasis in achieving a useful (preserved) end product. The basic stages of freezing a liquid material and then heating the frozen material under vacuum to effect moisture removal are preferable to earlier attempts at food preservation which often involved dehydration by hot air convection methods.

Dried product quality is of prime importance in terms of nutritional value and appearance to the consumer. It is undesirable to damage the product during the processing and removal of liquids from substances. Hot air convection drying often results in product shrinkage and other detrimental effects.

Batch unit operations have been used in the freeze drying industry although batch processing has a number of disadvantages which include; longer product processing durations, longer residence times at each stage of processing (often as manual labour is required to transfer partially processed substances from one stage to the next), poorly optimised processing equipment often with excess capacity, increased set-up times, reduced control of the process due to increased likelihood of human error (due to lack of automation), and low throughput. Batch systems are typically used for small production runs or where a need for process flexibility is required.

In conventional (batch freeze) drying processes at least one liquid feed is poured into shallow tray(s) (product thickness typically varies between 10 and 20mm), which are then placed on shelves in the freeze dryer. The door to the batch freezer is closed and the product is frozen. After the product has frozen, the trays are heated and the ice is slowly sublimed. The sublimed vapour is condensed on refrigerated coils. Once it is assumed that the product is dry, the product is removed manually. The product exits a conventional freeze-dryer as a brittle cake, and usually requires a separate granulation stage before it can be further processed.

This method of drying a moisture laden substance by freezing it then subliming off as much excess moisture as possible to produce a dried product is primarily used within industries where substances need to be dried, but are unable to withstand even moderate temperatures, for example some foodstuffs or pharmaceuticals can be damaged or affected by heat.

It is not unusual for this combined freezing and drying process to take 48 hours or more. This is undesirable if heat-sensitive materials are being dried. Also, if the vacuum is lost in a conventional batch freeze-dryer, melting and glass formation may occur, and it is possible that the entire load of product may be lost, that is the product may not be a useable quality product or able to be sold. In addition, the loading and unloading process is susceptible to product contamination due to exposure, and wastage from spillage of the shallow trays.

Therefore, a system which enables rapid freeze drying of a moisture laden substance and which produces a suitably formed end product, such as a powder, is desirable. As yet no truly continuous or semi-continuous freeze drying processes have been effectively developed by the drying industry.

An apparatus able to minimise potential contamination, reduce liquid feed spillage and which may increase the liquid throughput would be advantageous to the drying industry. If a liquid feed is able to be freeze dried which does also not require a necessary pre-treatment liquid feed cooling stages or subsequent dried product granulation stages, then significant problems with conventional batch freeze drying may be overcome.

In more recent times, the stages of freezing and drying have been further developed to include various steps such as reducing the physical size of substances to be treated, as well as graded temperature control during the freezing and drying stages. However, in practice highly controlled drying has often been difficult to implement effectively as the substances being dried are often in static tray type arrangements and therefore some substance is likely to be affected or heated more so than other parts of the substance, therefore resulting in over-dried or overheated and consequently damaged product. It would therefore be advantageous to utilise a drying stage with the provision of improved heat transfer conditions to substances, such a stage being preferably coupled to a freeze dry process.

It is therefore an object of the present invention to provide a dryer apparatus and/or a drying process or method which goes at least some way towards addressing the foregoing problems or to at least to provide the industry with a useful choice.

All references, including any patents or patent applications cited in this specification are hereby incorporated by reference. No admission is made that any reference constitutes prior art. The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinence of the cited documents. It will be clearly understood that, although a number of prior art publications are referred to herein, this reference does not constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country.

It is acknowledged that the term 'comprise' may, under varying jurisdictions, be attributed with either an exclusive or an inclusive meaning. For the purpose of this specification, and unless otherwise noted, the term 'comprise' shall have an inclusive meaning - i.e. that it will be taken to mean an inclusion of not only the listed components it directly references, but also other non-specified components or elements. This rationale will also be used when the term 'comprised' or 'comprising' is used in relation to one or more steps in a method or process.

Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only.

DISCLOSURE OF INVENTION

Accordingly, in a first aspect, the invention broadly consists of a method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein comprising the steps:

holding a chamber at a temperature and pressure below the triple-point of the liquid substance,

injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

collecting the FLS portion as a layer on a surface, and

conveying the collected FLS portion on the surface,

wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface.

A "liquid substance" is used herein and refers to a substance which has liquid flow-like properties, but has constant weight/mass in a first state. The weight/mass of the liquid substance can be altered by removing components from the liquid substance, for example by evaporating liquids from the substance, leaving a more concentrated liquid substance. Such liquid substances may include liquids having particles or solids suspended or dissolved in a solution, as well as compounds which can exist in a liquid state, such as or similar to water or oil.

Preferably, the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

Preferably, the FEL is condensed by a condensing device.

Preferably, the FEL is removed from said chamber.

Preferably, injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS.

Preferably, injecting and/or atomising of the liquid substance is performed by one or more nozzles.

Preferably, the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface.

Preferably, the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion.

Preferably, the predetermined particle size is substantially 500 µm or more.

Preferably, the predetermined particle size is less than substantially 500 µm.

Preferably, the predetermined particle size is less than substantially 200 µm.

Preferably, the surface conveys the FLS portion away from the one or more nozzles.

Preferably, the method includes the step of exposing the collected layer of FLS portion to a heating means thereby substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product.

Preferably, the SEL portion is condensed by a condensing device.

Preferably, the SEL portion is removed from the chamber.

Preferably, the product is removed from the conveying surface by a product removal device.

Preferably, the product is removed from the chamber via the outlet port.

Preferably, the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein.

Preferably, the substance is selected from one or more of the following: a slurry of coffee, liquid milk, fruit and/or vegetable juices.

In a second aspect, the invention broadly consists of an apparatus for drying or concentrating a liquid substance comprising:

a chamber and a chamber pressure reduction device, and

one or more injection ports through which the liquid substance is injected to the chamber,

a collection surface which collects a frozen liquid substance portion of the liquid substance,

wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled.

Preferably, the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

Preferably, the FEL portion is condensed by a condensing device.

Preferably, said FEL is removed from said chamber.

Preferably, the one or more injection ports comprises at least one spray or atomisation nozzle.

Preferably, the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle size.

Preferably, the pressure reduction device is a gas evacuation pump.

Preferably, the condensing device is a cooled coil or coils.

Preferably, the condensing device is cooled with a refrigerant.

Preferably, the collection surface includes one, or a combination of, the following conveyors: a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray.

Preferably, the angled tray and/or vibrating tray includes a reduced friction surface.

Preferably, the reduced friction surface comprises polytetrafluroethylene (PTFE).

Preferably, the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports.

Preferably, the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product.

Preferably, the heating means is one or a combination of the following energy sources: infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface.

Preferably, a product removal device is employed to remove the product from the collection surface.

Preferably, the product removal device is a scraping means and/or a brushing means.

Preferably, the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port.

Preferably, the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port.

Preferably, the product is removed via a chamber outlet port.

Preferably, the chamber is held at a pressure of substantially 611.3 Pa or less.

In a third aspect, the invention broadly consists of a drying process for a liquid substance comprising the steps of:

atomising the liquid substance,

cooling the atomised liquid substance (ALS) to initiate a phase change,

conveying the ALS into a drying chamber under a vacuum,

heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber.

Preferably, the liquid substance is chilled from an initial temperature prior to atomising.

Preferably, the ALS conveyed into the drying chamber is also conveyed through the drying chamber.

Preferably, the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

Preferably, the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature.

Preferably, atomising the liquid substance achieves a predetermined particle size.

Preferably, the predetermined particle size is substantially 500 μm or more.

Preferably, the predetermined particle size is less than substantially 500 μm.

Preferably, the predetermined particle size is less than substantially 200 μm.

Preferably, a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

Preferably, chilling is undertaken with direct or indirect contact with a cold fluid.

Preferably, said cold fluid is air.

Preferably, said cold fluid is substantially 0°C or less.

Preferably, said cold fluid is less than substantially -20°C.

Preferably, said spray freezer operates in a counter current configuration.

Preferably, said spray freezer operates in a co-current configuration.

Preferably, the ALS is reduced to a temperature below its eutectic temperature.

Preferably, the ALS is conveyed to a separator.

Preferably, said ALS is conveyed pneumatically to a separator.

Preferably, said separator is a gas-solid separation device.

Preferably, said gas-solid separation device is a cyclone.

Preferably, the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step.

Preferably, one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet.

Preferably, separated solids from said gas-solid separation device enter a vacuumous drying chamber.

Preferably, the vacuum of said vacuumous drying chamber is created by a pressure reduction device.

Preferably, the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

Preferably, the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

Preferably, said ALS is conveyed via a surface.

Preferably, said ALS is conveyed via a vibrating surface.

Preferably, said vibrating surface is a vibrating tray.

Preferably, said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

Preferably, said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation.

Preferably, a vapour produced by sublimation is removed from the drying chamber.

Preferably, vapour produced by sublimation is removed from the drying chamber by one or more condensers.

Preferably, the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

Preferably, said temperature gradient is provided by an energy source.

Preferably, said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

Preferably, atomising the liquid substance achieves a predetermined particle size.

Preferably, the predetermined particle size is substantially 500 µm or more.

Preferably, the predetermined particle size is less than substantially 500 μm.

Preferably, the predetermined particle size is less than substantially 200 μm.

Preferably, a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber.

Preferably, a product produced is substantially reduced is free of liquid compared to the liquid substance.

In a fourth aspect, the invention broadly consists of an apparatus for a drying process for a liquid substance comprising:

an atomiser capable of atomising a liquid substance,

a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,

a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,

an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and

a collector capable of collecting the dried ALS.

Preferably, a chiller capable of chilling a liquid substance from an initial temperature to a lower temperature is provided prior to the liquid substance atomiser.

Preferably, the apparatus includes a conveyor capable of conveying the ALS through the drying chamber.

Preferably, the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation.

Preferably, the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber.

Preferably, atomising the liquid substance achieves a predetermined particle size.

Preferably, the predetermined particle size is substantially 500 μm or more.

Preferably, the predetermined particle size is less than substantially 500 μm.

Preferably, the predetermined size is less than substantially 200 μm.

Preferably, a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

Preferably, said cold gas is air.

Preferably, said cold gas is substantially 0°C or less.

Preferably, said cold gas is less than substantially -20°C.

Preferably, the vacuum of said vacuumous drying chamber is created by a pressure reduction device.

Preferably, the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

Preferably, the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

Preferably, said ALS is conveyed via a vibrating surface.

Preferably, said vibrating surface is a vibrating tray.

Preferably, said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

Preferably, ALS is conveyed through a temperature gradient to substantially effect sublimation.

Preferably, vapour produced by sublimation is removed from the drying chamber by one or more condensers.

Preferably, said temperature gradient is provided by an energy source.

Preferably, said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

In a fifth aspect, the invention broadly consists of a drying chamber which comprises;

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray.

Advantageously, the present invention provides an improved method/process of treating a liquid substance to form a dryer or at least more concentrated form of the liquid substance (in the form of a 'product).

In preferred embodiments, the present invention is able to be operated on a semi-continuous basis, or at least which will allow a greater throughput of liquid substance than compared with prior art drying systems. The rate of conveyance of atomised liquid substance (ALS) is sufficient to increase efficiencies of drying/concentrating and/or which helps to reduce damage to the product.

BRIEF DESCRIPTION OF DRAWINGS

Further aspects of the present invention will become apparent from the following description which is given by way of example only and with reference to the accompanying drawings in which:

<u>Figure 1</u> illustrates one embodiment of a possible dryer configuration according to the present invention in the third and fourth aspects.

<u>Figure 2</u> is a process flow diagram of one possible embodiment of the present invention according to the first and second aspects;

<u>Figure 3</u> is a top view of of the drying chamber configuration shown in Figure 2,

<u>Figure 4</u> is a side view of the drying chamber configuration shown in Figures 2 and 3, and

<u>Figure 5</u> is an end view of the drying chamber configuration shown in Figures 2 to 4.

BEST MODES FOR CARRYING OUT THE INVENTION

The present invention may now be described with reference to Figures 1 to 5, and the accompanying description below.

The present invention relates to process for freeze drying or dehydrating/concentrating liquid substances. The process is applicable to liquids having a solid substance in suspension (for example milk) or to liquid solutions in which a substance has been dissolved. Tea, coffee, fruit juice, pharmaceuticals and nutraceuticals could also be processed using this spray freeze dryer.

Freeze drying is a useful preservation technique and among many other products, the following may be dried in this manner; instant coffee, vegetables for dried soup mixes, mushrooms, herbs, spices, cheese starter cultures, shrimp, fruits for ready-to-eat breakfast cereals, nutraceuticals, pharmaceuticals and agriceuticals.

Some specific end users of freeze dried products may include the production of military and/or space rations as well as light weight camping foods containing vegetables, meat, fish, and fruits. The freeze drying process may generally have the following advantages; low thermal damage, good retention of volatile flavours, good vitamin retention, rapid product rehydration, low product shrinkage, long product storage life – if suitably packed, retention of biological activity. Although there are also some disadvantages associated with freeze drying substances may be most notably; high drying cost, damage to certain products by initial freezing, rapid deterioration unless packed and maintained at low humidity, friability (i.e. crumbles easily), pre-treatment sometimes necessary (e.g. carrots) to avoid colour loss.

A process 1 as generally outlined in the third aspect, and apparatus as broadly defined by the fourth aspect described above, comprises holding a chamber 2 at a temperature and pressure below the triple-point of a liquid substance 3, injecting the liquid substance 3 into the chamber 2 thereby generating a frozen liquid substance portion 4 and a first evaporated liquid substance portion 5A.

The first evaporated liquid substance portion 5A may be condensed by condensing means 6, whilst at the same time collecting the frozen liquid substance portion 4 as a layer 7 on a collection surface 8.

The frozen liquid substance portion 4 collected as the layer 6 is conveyed along the collection surface 8. The surface 8 conveys the frozen liquid substance portion layer 7 at a rate which controls the thickness of the layer of collected frozen liquid substance portion collected.

The condensed first evaporated liquid substance portion may be removed from the chamber 2. In order for any water or evaporated liquid substance portion to be removed from the gaseous phase in the chamber, it can be condensed on the coils. In order for evaporated portion to be removed from the condensing coils, the coils can be returned to pressures above the triple point so that the ice will melt. Therefore, in order to achieve substantially continuous or quasi-continuous operation two or more sets of condensation coils may be used; while one is frosting up, the other or remaining coils may be isolated from the drying chamber by closing a pressure seal of some sort, returning to ambient pressure, and defrosted.

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Liquid substance 3 in a holding tank T may be sprayed into the chamber 2 in order to achieve a pre-determined size of frozen liquid substance portion particles, and this may be performed by one or more nozzles 10.

Thickness of the frozen liquid substance portion (particles) collected on the collection surface is determined by the rate (metres per second) of conveyance by the collection surface 8 which is in form of an endless conveyor (not shown). The thickness of this monolayer of particles will also be determined by the particle sizes generated by injection of the liquid substance into the chamber and/or of liquid substance injection by the nozzle(s) 10.

Desirably the surface conveys the frozen liquid substance portion away from the point of liquid substance injection, or the position of the one or more nozzles, at a rate which substantially achieves about the monolayer thickness of frozen liquid substance portion upon the collection surface 8.

The collected frozen liquid substance portion can then be exposed to a heating means 11 which may induce at least some sublimation and thereby generation of a second evaporated liquid substance portion 5B and a product 12.

The second evaporated liquid substance portion 5B can also be condensed by the condensing means 6 and removed from the chamber 2.

The product 12 (in a state advantageously containing less liquid than at the holding tank, T, state) may be removed from the collection surface 8 by a product removal means 13 then evacuated from the chamber 2 via the outlet port 14.

In a further embodiment of the process substantially as described above, an apparatus may be configured to generate a product 12 which is drier or more concentrated, than that of the liquid substance 3 in the holding tank, T.

A chamber 2 is held at a pressure at least below the triple-point pressure of the liquid substance by a pressure reduction means, such as a gas evacuation pump 15.

One or more injection ports, such as nozzles 10, may be employed through which the liquid substance 3 can be sprayed. Upon injection (or spaying) of the liquid substance 3 into the chamber 2, a frozen liquid substance portion 4 and a first evaporated liquid substance portion 5A may be generated.

A collection surface 8 is provided to substantially collect the generated frozen liquid substance portion 4 of the liquid substance 3.

A condensing means 6 may be employed to condense the first evaporated liquid substance portion 5A, and the condensate may be subsequently drained and removed from the chamber 2.

An outlet port 14 from the chamber may be used to evacuate a drier or more concentrate form of the liquid substance 3, in the form of a product 12.

The frozen liquid substance portion 4 can be accumulated as a layer 7 on the collecting surface and conveyed away from the one or more injection ports, towards the outlet port, at a rate which allows the accumulation of not more than substantially a monolayer thickness of frozen liquid substance portion.

The first evaporated liquid substance portion 5A can be condensed by the condensing means 6 and removed from the chamber. The condensing means 6 is a cooled condensation coil or coils. The condensing means is cooled with a refrigerant, supplied from the refrigeration unit 16.

The one or more injection ports comprise a spray or an atomisation nozzle or nozzles. The nozzle or nozzles substantially determine the size of the frozen liquid substance portion generated.

The collection surface includes one or a combination of the following conveyors: a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray. The angled tray and/or vibrating tray include a reduced friction surface comprising polytetrafluroethylene.

The layer of frozen liquid substance portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports which may substantially induce sublimation of the frozen liquid substance portion layer 7 upon the surface 8 to form a second evaporated liquid substance portion 5B and a product 12.

The heating means is one or a combination of the following energy sources: infra-red lamps, halogen lamps, incandescent lamps, microwaves or ohmic heating of the surface 8 itself which may act directly upon the frozen liquid substance portion 4. The heating means may also operate upon the frozen particles indirectly via heat transfer from the surround internal chamber equipment, such as heat transfer through the collection surface 8, or via radiation or reflection of energy from the surrounding chamber walls

A product removal device can be employed to remove the product from the collection surface which may have stuck or adhered to the surface. Such a product removal device may be a scraping means and/or a brushing means.

A scraping means may substantially contact the surface 8 with the product 12 thereon and move the product off the surface toward the outlet port 14, whereas a brushing means may be in the form of a rotating brush or fixed brush which also substantially contacts the surface with the product thereon and moves product toward the outlet port. Once the product is removed from the chamber it may then be packaged or further processed (such as a granulation stage, or vacuum packaging to maintain the quality of the product).

The liquid substance 3 may be supplied under pressure to the injection point 10 via a pump 17, with the liquid substance passing into the depressurised environment of the chamber 2 through a series of valves 18A.

The frozen portion 5A of the sprayed or atomised injected liquid substance 3 is advantageously directed to land and be collected upon a surface 8 which then conveys the frozen portion 4 away from the injection nozzle(s) 10.

The surface 8 may promotes travel of the frozen portion 4 away from the injection nozzle(s) by a either use of or a combination of conveyor surface options. These surface options are provided which allow the surface collected frozen portion 4 to preferably not accumulate to greater than about a monolayer thickness of frozen portion 4. The thickness or layer 7 of the frozen portion 4 upon the surface 8 directly determines the rate of sublimation (drying) during exposure to the heating means 11.

Alternatively, an optimal thickness for rapid sublimation (drying) of the frozen portion may be determined by actively measuring the thickness of the layer on the surface, for example using light reflection techniques. Once the thickness of the layer being sprayed onto the surface is determined, the conveyer rate of removal of particles away from the injection port(s) can be altered to match the liquid substance inlet spray rate, or to optimise the liquid substance throughput (and product production). The spray rate or throughput of liquid substance may also be altered to be sympathetic with the rate of conveying of particles along the surface.

Once the frozen portion 4 is exposed to the heating means 11, sublimation is preferably induced thereby resulting in additional liquid removal from the now frozen liquid substance. The substantially reduced moisture content substance, which may be referred to as the 'product' 12, can then be removed from the conveying surface. The product can then be collected and removed from the chamber 2 via an outlet port 14, which includes one or more valves 18B (or air locks) to prevent loss of chamber vacuum.

As the chamber 2 is held at a vacuum, a vacuum pump 15 is required to obtain desired pressures in the chamber, for example, the triple-point of water is 611.3 Pa (or 4.585 mmHg, or 0.0887 psi). The pressure of the chamber must never be higher than the triple-point pressure of water, however, depending on the product, certain temperature constraints must be met to avoid glass formation and freezing point depression associated with the freeze concentration effects that occur as the product dries. Some products must be kept below their eutectic temperature while for others the glass transition temperature is critical. Since the pressure in the vessel will determine the sublimation temperature and hence the product temperature, often the process will have to operate at pressures significantly below 611.3 Pa, for example, coffee: -21°C and 94 Pa; fruit juices -30°C and 38 Pa. Of course, it is recognised that this technology may be applied to most liquid substances containing particles in suspension or a material dissolved therein. A person skilled in the art of thermodynamics would understand the relationship of pressure and temperature, and how the chamber conditions determine the quantity of flash evaporation (generation of the first evaporated liquid substance portion) and resulting temperature of the frozen liquid substance portion formed.

The chamber conditions may be varied to optimise the level of throughput and/or level of freezing occurring in the chamber.

As the liquid feed may be fed through a nozzle directly into the vacuum chamber, the nozzle and feed pressure are chosen such that a stable spray-jet is maintained. In a sub-triple point environment, a material cannot exist in the liquid state, meaning that some of the liquid is forced to vaporise almost instantaneously, causing the remaining liquid to freeze. Since the enthalpy of fusion duty does not need to be supplied by an external energy source, it is effectively "free". In addition, as a result of the same process, approximately one seventh of the sublimation duty is also free. This means that for a given load of product, the spray freeze dryer will require at least 10% less energy than a conventional batch freeze dryer.

The product is frozen into very fine particles, whose maximum size is limited by the surface tension and vapour pressure (typically 100μm or less). The frozen particles land on a heated tray or belt where it dries as moisture sublimes off and is condensed by the condensing coils as it is conveyed along the length of the dryer. Freeze drying times may be estimated from the following equation (Fellows, 1997):

$$t_d = \frac{x^2 \rho \Delta M \lambda_s}{8 k_d \Delta T}$$

Where: t_d = drying time (s), x = product layer thickness (m), k_d = thermal conductivity of dry product (W/mK), ρ = bulk density of product (kg/m³), ΔM = change in moisture content (dry basis), ΔT = temperature driving force (°C) and λ_s = enthalpy of sublimation (J/kg). This equation shows that the drying time is proportional to the square of the thickness of the layer of the product on the drying surface.

The present invention of the second aspect can be referred to as a spray freeze drying process in a quasi-continuous process, and may be integrated into a continuous production line, which would reduce the labour requirements associated with its operation, and could also eliminate the risk of exposure or product contamination. In addition, the need for a subsequent granulation stage is removed, since the dried product exits the dryer in powdered form.

A polytetrafluroethylene (PTFE) coated surface such as a belt conveying system can be attached to a variable-speed drive, allowing the residence time of the product on the belt to be varied between about 1 and 12 minutes depending on exposure to the heating means required to sublime off a desired amount of moisture. The length of the belt may be about 2m and the belt speeds may be varied between about 0.17m/min and 2m/min. The product can be sprayed through multiple nozzles, for example 4 nozzles in parallel, in order to distribute the product evenly across the width of the belt (which may be about 0.5m).

It is anticipated that the majority of particle sizes will be in the region of 10 to 100μm, and therefore, since it is likely that the product will adhere to the surface (or belt) even with the PTFE (polytetrafluroethylene) coating, a removal means such as a knife-edge, or brush or scraping means arrangement can be employed at the end of the conveyer ((or any position along the surface). If the scraper or knife-edge is not effective enough, a PTFE or other type of material brush can also be used to scrape the particles off. The brush arrangement may cause aggregation of the particles, which may or may not be beneficial, depending on the down-stream handling intended for the particular product.

The feed nozzle 10 can protrude directly into the vacuum chamber 2. The pressure within the vacuum chamber 2 is maintained below the triple point pressure of the liquid 3 and therefore

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as the liquid substance 3 exits the nozzle 10 some of the water is evaporated 5A instantaneously ("flashed") as a result of the pressure drop below the liquid's triple point pressure. The evaporation process removes substantial amounts of heat from the droplet with the result that the remaining moisture in the product is frozen. Based on the relative magnitudes of the latent heats of vaporisation (≈2250 kJ/kg) and fusion (≈333 kJ/kg), 1 gram of evaporated moisture would freeze approximately 6.5 grams of remaining water 4. Remaining moisture in the frozen liquid substance portion 4 may optionally be removed by an subjecting it to a heating means or energy source such as an infra-red, halogen lamp or other energy source 11 within the vacuum chamber.

Potential advantages of this type of arrangement would also include the significant reduction in external energy (electricity) requirement as almost no electricity is required for freezing the product as well as the associated reduction in the tray-heating electricity requirement; and the difficulties associated with the conveyance of the frozen product (caking, compacting, agglomeration, thawing/re-freezing) can be largely be removed.

The spray freeze drier involves less equipment (and therefore a reduced capital cost) due to its mechanically simpler design, and there is the potential to have much greater control of the lateral distribution of the product on the drying tray (because the spray pattern may be controlled whereas before, the frozen product simply dropped onto the tray).

Some of the major advantages of spray-freeze drying over conventional batch freeze drying are the greatly reduced drying times (a matter of seconds compared to a matter of hours); minimises possibility of damage to heat-sensitive materials; about a 10% reduction in energy costs due to the novel freezing process, which is effectively free of cost, and the need for subsequent granulation stage, which is often costly and can result in product loss is substantially removed.

By utilising this quasi-continuous process exposure to contaminants is avoided and the spray freeze dryer is able to be integrated into a continuous production line, and is much less labour intensive than conventional freeze driers.

In further aspects of the present invention as broadly defined by the first, second and/or fifth aspects, there is provided a drying process and apparatus as illustrated by a flow diagram of the improved continuous spray-freeze-drying process is shown in Figures 2, 3, 4 and 5. A liquid substance such as coffee (not shown) is chilled from its initial temperature to some lower temperature in a chiller 21. This step is optional. The liquid feed is directed under pressure via a pump (not shown), atomised through a high pressure nozzle (not shown) into a freezing chamber 22. Ideally, the particle size of the atomised liquid substance should be less than substantially 200-300 micron (µm). The feed is introduced into the freezing chamber 22 with a co-current flow of cold air (-20°C or less) and atomised and frozen to produce an atomised frozen substance (ALS). The ALS is quickly frozen below its eutectic temperature to prevent melting. The frozen ALS is pneumatically conveyed by the cold air stream to the spray freezer to a separating cyclone 23, where frozen ALS exits the bottom of the cyclone 23 and the outlet air stream exits the top of the cyclone 23. The outlet air stream from the spray freezer must be below the eutectic temperature of the frozen ALS. A fan or blower 24 blows the outlet air stream from the cyclone through a refrigeration system, in this instance an evaporator 25, where the air is cooled back down to its initial temperature before returning to the spray freezing chamber 22.

The frozen product then falls by gravity into a termination chamber (not shown) attached to the bottom of the cyclone. A vacuum or air lock 27 allows the product to pass from the bottom of the cyclone into the drying chamber 28 without interruption to the vacuum inside the drying chamber. The termination chamber and air lock are cooled with cold air from the refrigeration system, in this instance a cooler 26 to prevent the frozen ALS from melting. The drying chamber is maintained at an absolute pressure of substantially 200-400 micro-meters of Hg with a vacuum pump 212. Frozen ALS falls onto an inclined pneumatic vibrating tray 29 which conveys drying product towards the discharge end of the dryer. To effect sublimation and freeze drying, the tray is heated by a radiant and/or conduction heat source 210 placed underneath the tray 29. The surface temperature of the tray is controlled down its length to ensure complete drying without damaging the product (not shown). The dried ALS (product) exits through the discharge vacuum lock 213. Two condensers 211 are required to run the system continuously. One condenser would be operating normally during drying, while the other would be isolated from the drying chamber and would be defrosting.

An improved drying process for a liquid substance, according to the first aspect includes one or more of the steps of chilling the liquid substance from an initial temperature, atomising the liquid substance so that a predetermined particle size is achieved, freezing the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which is held under a vacuum, heating the frozen ALS so as to effect sublimation and freeze drying and then collecting the dried ALS from the chamber.

A liquid substance may be defined to be any substance containing a liquid. These may for example include, milk, coffee, liquor, pharmaceuticals, nutraceuticals, function food, agriceuticals, or any other substance which has a moisture content. The amount of liquid in a substance need only be minimal to allow over a period of time degradation of the substance, therefore removal of any "free" liquid is desirable.

The conveying of ALS to a drying chamber may be achieved by any suitable conveying methods/apparatus. For example, these may include pneumatic methods, mechanical methods, electrical or gravity assisted conveying methods may also be suitable.

In another embodiment of the first and/or second aspect preferably there is provided an improved drying process for a liquid substance which may include one or more of the steps of chilling the liquid substance from an initial temperature, atomising the liquid substance so that a predetermined particle size is achieved, freezing the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which is held under a vacuum, conveying the ALS through the drying chamber, and heating the frozen ALS via a temperature gradient while it travels through the drying chamber so that the temperature grading can effect sublimation and drying whilst preventing or minimising any heat damage occurring to the ALS as it passes through the chamber to a collection point of the dried ALS.

The step of chilling a liquid substance can take place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature. For example, this may mean that the liquid substance is chilled from ambient conditions and/or a storage condition facility temperature to a lower temperature. This chilling step is optional, however it may assist and reduce the cooling loading required by the spray freezer.

The chilling may be achieved by any suitable method or apparatus that can reduce the temperature of a substance from an initial temperature to a lower temperature. Preferably chilling may be achieved by any suitable heat transfer unit, these may for example be refrigerators, plate heat exchangers, shell and tube heat exchangers, heat pumps, cool air convection apparatus, gas-liquid cooling towards and other such suitable cooling apparatus.

The spray freezer is an especially important aspect of the continuous processing configuration in which rapid temperature reduction to below the substance eutectic temperature is desired, as well as atomisation of the liquid substance. Preferably, atomisation of the liquid substance may be induced by a variety of feed devices such as, single-liquid nozzle (pressure type), two-liquid nozzle (pneumatic type), centrifugal (spinning disc), ultrasonic nozzles and various other rotary atomisers and air atomisation techniques may be employed.

The cooling of the ALS may be achieved by the use of a cooler capable of freezing the ALS. Preferably the atomisation and cooling of the liquid substance both take place in a spray freezer.

There are a number of advantages to spray freezing, especially as particulates formed may be produced of specific or predetermined particle sizes and the specification of, or particulate quality, remains substantially constant throughout the entire spray freezer operation. Spray freezer operation is ideally continuous and adaptable for full automatic control, where response times are fast. Spray freezing is a useful application to both heat sensitive and heat resistant substances. However, it is appreciated that semi-continuous processing may also be required.

A phase change occurs in the ALS when the temperature of the ALS is reduced, that is in which liquids become frozen or reduced to below their eutectic temperature (i.e. solids). Liquids may be sublimated from the frozen ALS at a later state in the process once exposed to a temperature gradient and energy source.

Cyclones are one of the main methods used to separate gas-solid phases, and can provide efficient separation. Frozen ALS and gases are separated, with the gas being recycled to chill the liquid substance, and the frozen (solid) ALS being conveyed through the drying chamber. Sublimation is the phase change from frozen liquids (solid) to gas (liquid vapour) for the means of evaporation of liquid from the ALS. Ideally the sublimation and/or drying of the frozen ALS takes place on a surface. More preferably the surface may be a vibrating surface. Even more preferably, the vibrating surface is a vibrating tray in which the vibrations are produced by pneumatic and/or mechanical and/or electrical means. It is advantageous to induce and/or provide some vibration movement of the frozen ALS to be dried to enhance and promote improved heat transfer to the atomised particles.

The sublimation (i.e. drying of the ALS and "free" liquid removal) may be initiated and effected by the energy emitted from an energy source such as an infrared device, which the vibrating drying tray preferably enhances the heat transfer characteristics to the ALS and allows for a more uniform and controlled drying stage. The energy source capable of heating the ALS may be any suitable energy source for inducing sublimation of the frozen liquids of the ALS. Such suitable energy sources may for example include; infra-red emitting devices, microwaves, radiant heaters, convection heaters, and any device which provides enough energy to the ALS to induce sublimation of the frozen "free" liquids of the ALS.

The removal of liquids from substances to form a dried product by controlling air flow rates, temperature and pressure reduced the moisture content of substances assists in inhibiting microbial growth that may cause decay and spoilage. Moisture removal also reduces weight, which is of significant interest and consideration for shipping and transport.

Chemical pre-treatment of some substances may further aid the longevity of product shelf life. These pre-treatments may include substances to enhance preservation.

Often dehydration on an industrial scale requires a feed product to be reduced in size and/or particulated to allow enhanced processing characteristics. Freeze drying is the drying of material in a frozen state and preferably the drying stage takes places at an absolute pressure that may readily permit ice to undergo a phase change directly from solid to vapour. In the present invention freeze dried product is preferably substantially undamaged from the drying chamber stage.

In an improved drying process of the first and second aspects, the liquid substance may be atomised in a spray freezer to achieve a particle size of substantially 500 microns or more. Alternatively, the particle size achieved may be less than substantially 500 microns, or even less than substantially 200 microns. Atomisation of the liquid substance is desirable so that the atomised liquid substance may be rapidly reduced in temperature to below its eutectic temperature. Further, atomisation of the liquid substance and subsequent freezing allows for a particulate product to be processed which allows for enhanced drying control and heat transfer characteristics.

The apparatus or spray freezer may be of a counter current configuration, although it may also be of co-current configuration in which the spray freezer can utilise a cold gas to effect a phase change of the liquids in the ALS. The cold gas can effect a phase change causes freezing of liquids within the atomised liquid substance (that is, the ALS is reduced to a temperature below its eutectic temperature). The cold gas utilised by the spray freezer to effect the phase change may be cold air, although any suitable gas to effect a phase change of liquids contained within the ALS may be used.

The cold gas used may be substantially 0°C or less, even more preferably the cold gas may be less than substantially - 20°C.

The ALS once frozen/reduced to below its eutectic temperature can then be conveyed to a separator, and conveying may take place pneumatically. The separator can be a gas-solid separation device, for instance a cyclone.

The gas separated from the cyclone may be returned and/or refrigerated for use in the spray freezer. It can also be recycled to the spray freezer to reduce the cooling load on the evaporator. The evaporator is used to cool the gas which is used to induce a phase change of liquids in the ALS. However, any suitable gas cooling apparatus may be used, with an evaporator being used as an example in this case. Suitable cooling apparatus may include refrigerators, plate heat exchangers, shell and tube heat exchangers and other heat transfer units capable of cooling a gas to a temperature that can be supplied to a spray freezer to force a phase change of liquids in the ALS to the solid (frozen) state.

Preferably one or more vacuum and/or air locks are in place between the frozen ALS cyclone outlet and drying chamber inlet, or some other type of pressure seal device. It is advantageous to provide one or more pressure lock devices for maintenance of the vacuum within the drying chamber. Without the use of these pressure lock devices a suitable vacuum in the drying chamber may not be obtainable. Pressure locks such as valves may be particularly suitable, although any suitable device for sealing the vacuum drying chamber and ensuring the drying chamber maintains its vacuum may be utilised.

In further embodiments there can be a refrigeration system which is employed to maintain the frozen ALS at a temperature below its eutectic temperature during transport from the cyclone to the vacuumous drying chamber. It is advantageous that the frozen ALS remains at a temperature below it eutectic temperature to minimise any product degradation or deterioration that may occur if the ALS rises about its eutectic temperature.

The vacuum of the vacuumous drying chamber is created by a suitable pressure reduction device.

The vacuum created by such suitable pressure reduction devices may be substantially 600 micron meters Hg at select pressure or less. More preferably the vacuum created may be in the range of substantially 200-400 micro meters Hg absolute pressure.

Transport or conveying of the frozen ALS through the vacuum chamber can take place on a vibrating surface, where the vibrating surface is a vibrating tray. The vibrating tray may be pneumatically, electrically and/or via mechanical means driven for provision of the vibration to the tray.

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The vibrating tray conveys frozen ALS through a temperature gradient to effect sublimation, with the vapour produced by sublimation being removed from the drying chamber by one or more condensers or such like.

The temperature gradient across the drying tray is provided by an energy source, such as an infrared emitting device. However, the use of other energy sources are envisaged, for example Ohmic heaters and micro-wave devices.

The product produced by an improved drying process and apparatus is substantially free of liquid, otherwise substantially dried or at least substantially reduced in liquid content.

An apparatus for improved drying which facilitates one or more of the following steps of chilling a liquid substance from an initial temperature to a lower temperature, atomising a liquid substance so that a predetermined particle size may be achieved, cooling the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which may be held under a vacuum, and heating the frozen ALS so as to effect sublimation and freeze drying and then collecting the dried ALS from the drying chamber is desired. In an alternative embodiment the apparatus is defined as an apparatus for an improved drying process which may facilitate one or more of the steps of chilling the liquid substance from an initial temperature, atomising the liquid substance so that a predetermined particle size may be achieved, cooling the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which is held under a vacuum, conveying the ALS through the drying chamber and, heating the frozen ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation and drying whilst preventing or minimising any heat damage occurring to the ALS as it passes through the chamber to a collection point of the dried ALS.

It would be preferable but not necessarily essential that the steps of the process and methodology may be linked to form a continuous process. A continuous process may have a number of advantages.

In addition, a process which is configured to be run on a continuous basis may be preferable as a continuous system tends to be more easily optimised for productivity, higher throughput, reduced processing time per unit produced, improved quality as a result of optimised controls, a generally more efficient process, reduction of human handling and/or contact with substances which may contaminate equipment and/or substances is desirable, and the reduction of human manual labour preferably also reduce the likelihood of injury. Further, continuous processing may allow for equipment designed for an optimised solution for continuous processing and production of freeze dried products.

The vacuum drying chamber is designed to operate at absolute pressures in the range of 200-400 microns of Hg. The vacuumous state of the drying chamber allows for the preferential phase change (sublimation) of the frozen liquids to gas/vapour phase. The sublimation material (vapour) may be preferably removed by the use of a condenser. In a preferred embodiment the drying chamber is run continuously, and may be linked with additional spray freezers, separating devices (cyclones), packaging systems to be run in a batch, semi-continuous or continuous basis.

In a preferred embodiment of the present invention, the frozen ALS may fall onto an inclined pneumatic vibrating tray, (although other vibrating tray systems may be employed) to convey the ALS through a temperature gradient to effect sublimation and discharge of dried product at the end of the drier. The dried product exits through a discharge vacuum lock (which ensures the vacuum is maintained) into further processing stages. For example, further processing stages may include discrete packaging systems.

It is preferable that the surface temperature of the tray is controlled down its length to ensure complete drying without damaging of products.

Drying may be defined as the application of energy under controlled conditions to remove the majority of the water normally present in a substance by evaporation. The main purpose of dehydration is to extend the shelf life of substances by a reduction in water content.

Drying may cause a deterioration in the eating quality and nutritive value of the food. Water plays an important role in the stability of fresh, frozen and dried foods as it acts as a solvent for chemical, microbiological and enzymatic reaction.

A dryer may include a vibrating tray, a pressure reducing means, a material inlet port, a material outlet port, and a heat source. The vibrating tray provides a vibrating surface (upon which sublimation occurs) which conveys frozen ALS from the inlet material port to the outlet material port. The inlet material port is the entry point for frozen ALS into the dryer, and the outlet material port is the exit point of dried ALS (post sublimation).

A heat source is used to induce sublimation of frozen liquids from the frozen ALS along the length of the vibrating tray.

It is therefore advantageous to provide a series of treatment stages in which a liquid substance may be processed and substantially dried or have its liquid content reduced. The steps as described of optionally chilling a liquid substance, atomising the liquid substance, cooling the ALS to initiate a phase change of the liquids within the ALS, optionally separating the phase changed ALS from the cooling gas, conveying the phase changed ALS to a drying chamber, and removal of liquids from the ALS by sublimation may be referred to as a series of treatment stages to provide a substantially reduced liquid/moisture content substance.

However, it is also considered that the pre-treatment steps may be foregone and the liquid substance may be inserted directly to the vacuum chamber (which is held at a vacuum pressure which may be below the triple-point pressure of the liquid substance being processed). This direct injection may result in the flash evaporation of a portion of the liquid with the remainder of the liquid substance consequently being reduced in temperature such that frozen liquid substance particles result. These frozen particles may then be treated within the drying chamber to induce sublimation of remaining liquid in order to achieve a substantially drier liquid substance.

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The pre-treatment stages may be considered to enhance the overall process, although they may be foregone where direct injection of the liquid substance to the vacuum pressure drying chamber is an option.

Those skilled in the art will appreciate, understand and be able to calculate the sizings of pumps, heaters, motors, spray or atomisation nozzles required to achieve the present invention, and it is appreciated that there are numerous variables which will dictate or influence the sizings of these sorts of components. It should also be appreciated that design of a vacuum chamber will be within the realm of a person skilled in the art – however it is the combination and advantages conveyed above that make this invention suitable.

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope thereof as defined in the appended claims.

THE CLAIMS DEFINING THE INVENTION ARE:

1. A method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein including the steps of:
holding a chamber at a temperature and pressure below the triple-point of the liquid substance,
injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),
collecting the FLS portion as a layer on a surface, and
conveying the collected FLS portion on the surface,
wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface.

2. A method as claimed in claim 1, wherein the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

3. A method as claimed in claim 1 or claim 2, wherein the FEL is condensed by a condensing device.

4. A method as claimed in anyone of claims 1 to 3, wherein the FEL is removed from said chamber.

5. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS.

6. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising of the liquid substance is performed by one or more nozzles.

7. A method as claimed in any one of the preceding claims, wherein the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface.

8. A method as claimed in claim 7, wherein the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion.

9. A method as claimed in claim 8, wherein the predetermined particle size is substantially 500 μm or more.

10. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 500 μm.

11. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 200 μm.

12. A method as claimed in any one of the preceding claims, wherein the surface conveys the FLS portion away from the one or more nozzles.

13. A method as claimed in any one of the preceding claims, wherein the method includes the step of exposing the collected layer of FLS portion to a heating means thereby

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substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product.

14. A method as claimed in claim 13, wherein the SEL portion is condensed by a condensing device.

15. A method as claimed in claim 13 or claim 14, wherein the SEL portion is removed from the chamber.

16. A method as claimed in any one of claims 13 to 15, wherein the product is removed from the conveying surface by a product removal device.

17. A method as claimed in any one of claims 13 to 16, wherein the product is removed from the chamber via the outlet port.

18. A method as claimed in any one of the preceding claims, wherein the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein.

19. A method as claimed in any one of the preceding claims where the substance is selected from one or more of the following: a slurry of coffee, liquid milk, fruit and/or vegetable juices.

20. An apparatus for drying or concentrating a liquid substance comprising:
a chamber and a chamber pressure reduction device, and
one or more injection ports through which the liquid substance is injected to the chamber,
a collection surface which collects a frozen liquid substance portion of the liquid substance,
wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),
so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled.

21. An apparatus as claimed in claim 20, wherein the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

22. An apparatus as claimed in claim 20 or claim 21, wherein the FEL portion is condensed by a condensing device.

23. An apparatus as claimed in any one of claims 20 to 22, wherein said FEL is removed from said chamber.

24. An apparatus as claimed in any one claims 20 to claim 23, wherein the one or more injection ports comprises at least one spray or atomisation nozzle.

25. An apparatus as claimed in claim 24, wherein the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle

size.

26. An apparatus as claimed in any one of claims 20 to 25, wherein the pressure reduction device is a gas evacuation pump.

27. An apparatus as claimed in any one of claims 20 to 26, wherein the condensing device is a cooled coil or coils.

28. An apparatus as claimed in any one of claims 20 to 27, wherein the condensing device is cooled with a refrigerant.

29. An apparatus as claimed in any one of claims 20 to 28, wherein the collection surface includes one, or a combination of, the following conveyors: a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray.

30. An apparatus as claimed in claim 29, wherein the angled tray and/or vibrating tray includes a reduced friction surface.

31. An apparatus as claimed in claim 30, wherein the reduced friction surface comprises polytetrafluroethylene (PTFE).

32. An apparatus as claimed in any one of claims 20 to 31, wherein the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports.

33. An apparatus as claimed in claim 32, wherein the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product.

34. An apparatus as claimed in claim 32 or claim 33, wherein the heating means is one or a combination of the following energy sources: infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface.

35. An apparatus as claimed in claim 33 or claim 34, wherein a product removal device is employed to remove the product from the collection surface.

36. An apparatus as claimed in claim 35, wherein the product removal device is a scraping means and/or a brushing means.

37. An apparatus as claimed in claim 36, wherein the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port.

38. An apparatus as claimed in claim 36, wherein the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port.

39. An apparatus as claimed in any one of claims 33 to 37, wherein the product is removed via a chamber outlet port.

40. An apparatus as claimed in any one of claims 20 to 39, wherein the chamber is held at a pressure of substantially 611.3 Pa or less.

41. A drying process for a liquid substance comprising the steps of:

atomising the liquid substance,
cooling the atomised liquid substance (ALS) to initiate a phase change,
conveying the ALS into a drying chamber under a vacuum,
heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber.

42. A drying process as claimed in claim 41, wherein the liquid substance is chilled from an initial temperature prior to atomising.

43. A drying process as claimed in claim 41 or claim 42, wherein the ALS conveyed into the drying chamber is also conveyed through the drying chamber.

44. A drying process as claimed in any one of claims 41 to 43, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

45. A drying process as claimed in any one of claims 42 to 44, wherein the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature.

46. A drying process as claimed in any one of claims 41 to 45, wherein atomising the liquid substance achieves a predetermined particle size.

47. A drying process as claimed in claim 46, wherein the predetermined particle size is substantially 500 µm or more.

48. A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 500 µm.

49. A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 200 µm.

50. A drying process as claimed in any one of claims 41 to 49, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

51. A drying process as claimed in any one of claims 42 to 50, wherein chilling is undertaken with direct or indirect contact with a cold fluid.

52. A drying process as claimed in claim 51, wherein said cold fluid is air.

53. A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is substantially 0°C or less.

54. A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is less than substantially -20°C.

55. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a counter current configuration.

56. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a co-current configuration.

57. A drying process as claimed in any one of claims 41 to 56, wherein the ALS is reduced to a temperature below its eutectic temperature.

58. A drying process as claimed in any one of claims 41 to 57, wherein the ALS is conveyed to a separator.

59. A drying process as claimed in any one of claims 41 to 57, wherein said ALS is conveyed pneumatically to a separator.

60. A drying process as claimed in claim 58 or claim 59, wherein said separator is a gas-solid separation device.

61. A drying process as claimed in claim 60, wherein said gas-solid separation device is a cyclone.

62. A drying process as claimed in claim 60 or claim 61, wherein the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step.

63. A drying process as claimed in claim 61 or claim 62, wherein one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet.

64. A drying process as claimed in any one of claims 60 to 63, wherein separated solids from said gas-solid separation device enter a vacuumous drying chamber.

65. A drying process as claimed in any one of claims 41 to 64, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device.

66. A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

67. A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

68. A drying process as claimed in any one of claims 41 to 67, wherein said ALS is conveyed via a surface.

69. A drying process as claimed in any one of claims 41 to 68, wherein said ALS is conveyed via a vibrating surface.

70. A drying process as claimed in claim 69, wherein said vibrating surface is a vibrating tray.

71. A drying process as claimed in claim 69 or claim 70, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

72. A drying process as claimed in claim 70 or claim 71, wherein said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation.

73. A drying process as claimed in any one of claims 41 to 72, wherein a vapour produced by sublimation is removed from the drying chamber.

74. A drying process as claimed in claim 73, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers.

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75. A drying process as claimed in any one of claims 41 to 74, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

76. A drying process as claimed in any one of claims 41 to 75, wherein said temperature gradient is provided by an energy source.

77. A drying process as claimed in claim 76, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

78. A drying process as claimed in any one of claims 41 to 77, wherein atomising the liquid substance achieves a predetermined particle size.

79. A drying process as claimed in claim 78, wherein the predetermined particle size is substantially 500 µm or more.

80. A drying process as claimed in claim 78, wherein the predetermined particle size is less than substantially 500 µm.

81. A drying process as claim in claim 78, wherein the predetermined particle size is less than substantially 200 µm.

82. A drying process as claimed in any one of claims 60 to 81, wherein a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber.

83. A drying process as claimed in any one of claims 41 to 82, wherein a product produced is substantially reduced is free of liquid compared to the liquid substance.

84. An apparatus for a drying process for a liquid substance comprising:
an atomiser capable of atomising a liquid substance,
a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,
a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,
an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and
a collector capable of collecting the dried ALS.

85. An apparatus for a drying a drying process as claimed in claim 84, wherein a chiller capable of chilling a liquid substance from an initial temperature to a lower temperature is provided prior to the liquid substance atomiser.

86. An apparatus for a drying a drying process as claimed in claim 84 or claim 85, wherein the apparatus includes a conveyor capable of conveying the ALS through the drying chamber.

87. An apparatus for a drying a drying process as claimed in any one of claims 84 to 86, wherein the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the

temperature gradient can effect sublimation.

88. An apparatus for a drying process as claimed in claim 87, wherein the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber.

89. An apparatus for a drying process as claimed in any one of claims 84 to 88, wherein atomising the liquid substance achieves a predetermined particle size.

90. An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is substantially 500 μm or more.

91. An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is less than substantially 500 μm.

92. An apparatus for a drying process as claimed in claim 89, wherein the predetermined size is less than substantially 200 μm.

93. An apparatus for a drying process as claimed in any one of claims 84 to 92, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

94. An apparatus for a drying process as claimed in claim 93, wherein said cold gas is air.

95. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is substantially 0°C or less.

96. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is less than substantially -20°C.

97. An apparatus for a drying process as claimed in any one of claims 84 to 96, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device.

98. An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

99. An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

100. An apparatus for a drying process as claimed in any one of claims 84 to 99, wherein said ALS is conveyed via a vibrating surface.

101. An apparatus for a drying process as claimed in claim 100, wherein said vibrating surface is a vibrating tray.

102. An apparatus for a drying process as claimed in claim 101, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

103. An apparatus for a drying process as claimed in claim 84 or claim 102, wherein ALS is conveyed through a temperature gradient to substantially effect sublimation.

104. An apparatus for a drying process as claimed in claim 84 or claim 103, wherein vapour

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produced by sublimation is removed from the drying chamber by one or more condensers.

105. An apparatus for a drying process as claimed in claim 103, wherein said temperature gradient is provided by an energy source.

106. An apparatus for a drying process as claimed in claim 105, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

107. A drying chamber which comprises;
a device capable of vibrating a tray,
a suitable device for reducing the pressure in said chamber,
a material outlet port from said chamber, and
a heat source adapted to act upon said tray.

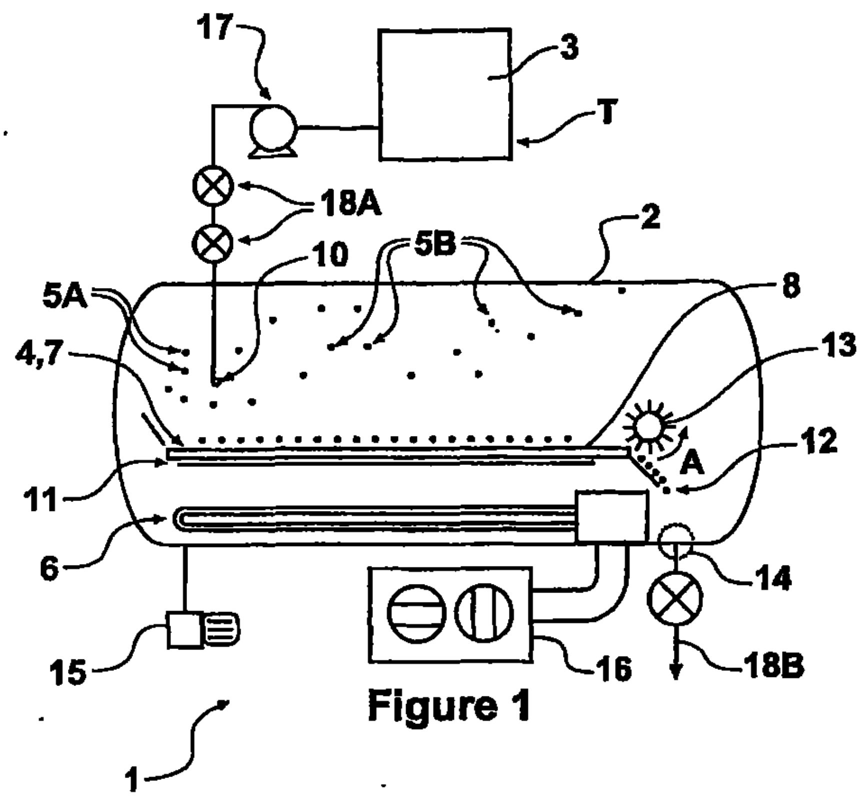

Figure 1

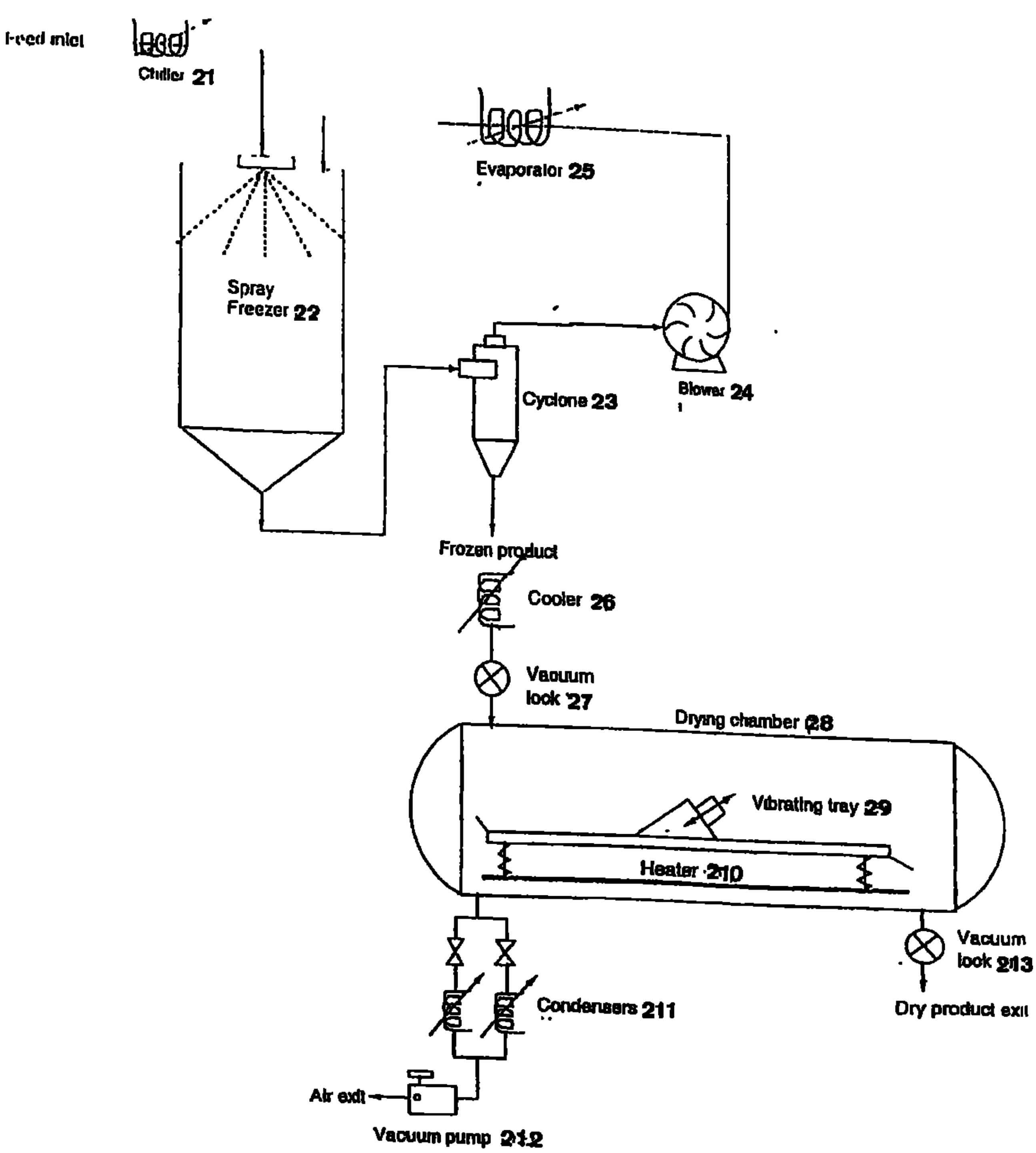
Feed inlet
Chiller 21
Spray Freezer 22
Evaporator 25
Blower 24
Cyclone 23
Frozen product
Cooler 26
Vacuum lock 27
Drying chamber 28
Vibrating tray 29
Heater 210
Vacuum lock 213
Dry product exit
Condensers 211
Air exit
Vacuum pump 212

FIGURE 2

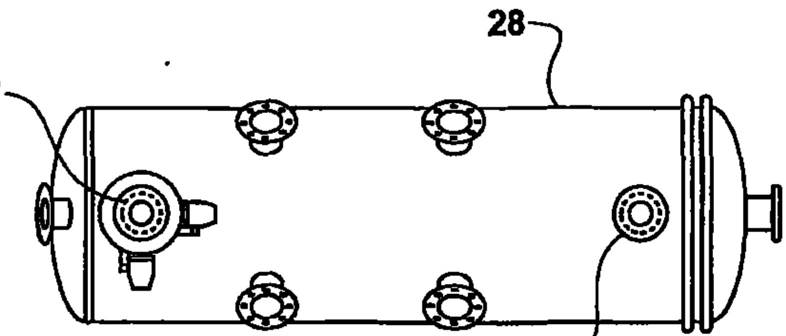

Figure 3

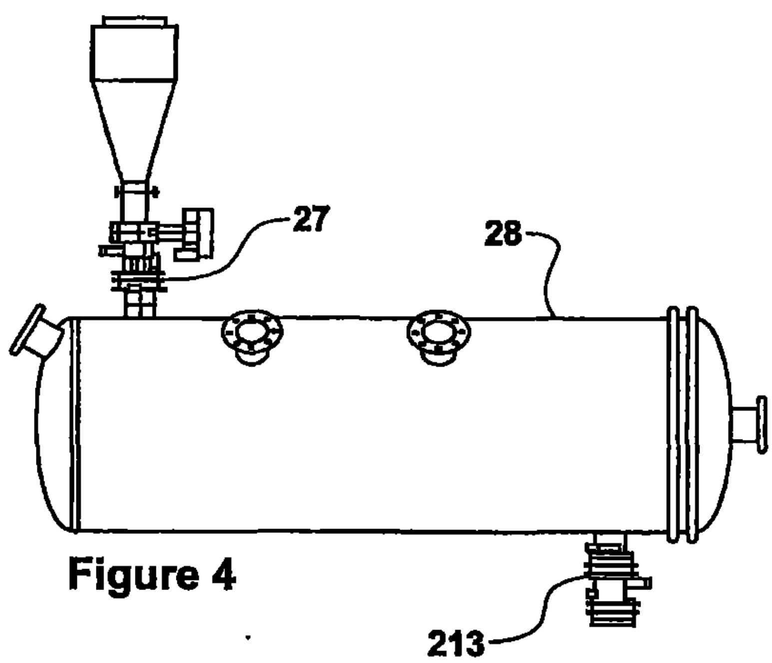

Figure 4

34 39

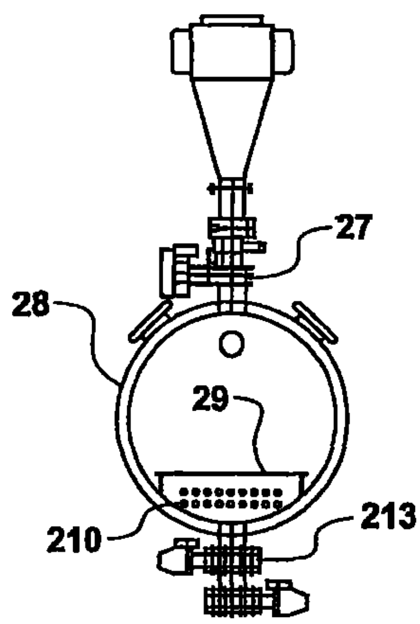

Figure 5

102 362
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/NZ2005/000089

A.	CLASSIFICATION OF SUBJECT MATTER
Int. Cl. 7:	B01D 1/18; F26B 5/06

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
DWPI: A23L 3/44, B01D 1/16, 1/18, 1/20, 9/04, F26B 5/06, 17/10; & Keywords - spray+, inject+, atomis+, atomiz+, freez+, froz+, dry+, concentrat+, (triple point)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	Derwent Abstract Accession No. 2004-576721/56, Class D13, J08, JP 2004232883 A (ULVAC CORP) 19 August 2004 Abstract	1-106
P, X	US 2004/0154317 A (SHEKUNOV ET AL) 12 August 2004 Whole document	1-106
X	Derwent Abstract Accession No. 2004-094584/10, Class D14 JP 2004012015 A (OKAWARA SEISAKUSHO KK) 15 January 2004 Abstract	1-106
X	Derwent Abstract Accession No. 88-358218/50, Class Q76 JP 63269969 A (HOUSE SHOKUHIN KOGYO) 8 November 1988 Abstract	1-106

[X] Further documents are listed in the continuation of Box C [X] See patent family annex

* Special categories of cited documents:
- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier application or patent but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed
- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 August 2005	25 AUG 2005

Name and mailing address of the ISA/AU	Authorized officer
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No (02) 6285 3929	Tharu Fernando Telephone No. (02) 6283 2486

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
X	US 4033048 A (VAN IKE) 5 July 1977 Whole document	1-106
X	Derwent Abstract Accession No. 82-41035e/20, Class J08, X25, Q76 SU 0848932 A (PRESERVES FOOD IND) 23 July 1981 Abstract	107
A	US 4810274 A (CHENG ET AL) 7 March 1989 Whole document	1-106

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/NZ2005/000089

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See supplemental sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims

2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest ☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

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INTERNATIONAL SEARCH REPORT

International application No
PCT/NZ2005/000089

Supplemental Box
(To be used when the space in any of Boxes I to VIII is not sufficient)

Continuation of Box No III **Observations where unity of invention is lacking (Continuation of item 3 of first sheet)**

This International Searching Authority found multiple inventions in this international application, as follows:

The international application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept. In coming to this conclusion the International Searching Authority has found that there are different inventions as follows:

1. Claims 1 to 106 are directed to a method of drying or concentrating a liquid substance whereby the liquid is sprayed into a chamber that is held at a temperature and pressure below the triple-point of the liquid, such that the sprayed liquid generates a frozen liquid substance and an evaporated liquid, the frozen liquid portion is collected and dried to sublime excess moisture and obtain dried solid particles. It is considered that holding the chamber at a temperature and pressure below the triple point of the liquid substance to such that frozen droplets are obtained comprises a first "special technical feature".

2. Claim 107 is directed to a drying chamber comprising a device capable of vibrating a tray, a device capable of reducing the pressure of the chamber, an outlet port and a heat source adapted to act on the tray. It is considered that the drying chamber as a whole comprises a second special technical feature.

Since the abovementioned groups of claims do not share any of the technical features identified, a "technical relationship" between the inventions, as defined in PCT rule 13.2 does not exist. Accordingly the international application does not relate to one invention or to a single inventive concept, a priori.

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member	
JP	2004232883	NONE	
US	2004154317	WO	2004071410
JP	2004012015	NONE	
JP	63269969	NONE	
US	4033048	NONE	
SU	0848932	NONE	
US	4810274	WO	9010838

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX

/02·362 45

(12) SOLICITUD INTERNACIONAL PUBLICADA SEGÚN EL TRATADO DE COOPERACIÓN INTERNACIONAL EN MATERIA DE PATENTES (PCT)

(19) Organización Mundial de Propiedad Intelectual
Oficina Internacional

(43) Fecha de Publicación Internacional
10 de noviembre de 2005 (10.11.2005)

PCT

(10) Número de Publicación Internacional
WO 2005/105253 A1

(51) Clasificación Internacional de Patentes[7] B01D 1/18, F26B 5/06

(21) No. Solicitud Internacional. PCT/NZ2005/000089

(22) Fecha de Presentación Internacional: 2 de mayo de 2005 (02 05 2005)

(25) Idioma de Presentación. Inglés

(26) Idioma de Publicación Inglés

(30) Datos relativos a la Prioridad·
529594 1 de mayo de 2004 (01 05 2004) NZ
529595 1 de mayo de 2004 (01 05 2004) NZ

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(75) Inventores/Solicitantes *(para US únicamente)* CARSON, James, Kenneth [NZ/NZ], c/- AgResearch Limited, Ruakura Campus, Private Bag 3123, 2020 Hamilton (NZ) HILL, Harold, Keith [NZ/NZ], 1845 Kakaramea Road, RD 10, 2020 Hamilton (NZ) GRAHAM, Donald, Michael [NZ/NZ], 22 Manor Place, 2020 Hamilton (NZ) SCHOU, Grant, Brian [NZ/NZ] C/- AgResearch Limited, Ruakura Campus, Private Bag 3123, 2020 Hamilton (NZ)

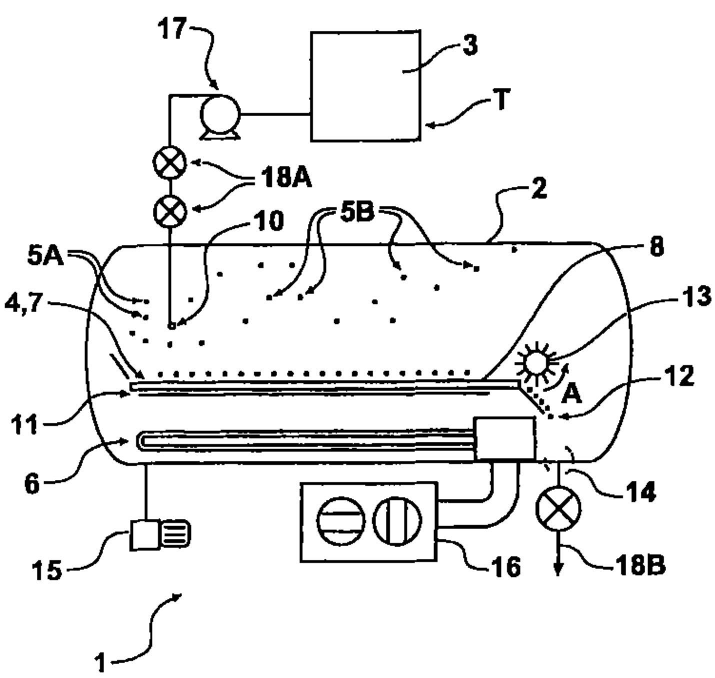
(74) Agentes. SIM, Jeremy et al , Private Bag 11907, Level 9, James & Wells Tower, 56 Cawley Street, Ellerslie, 1005 Auckland (NZ)

(81) Países designados *(a menos que se indique lo contrario para cada clase de protección disponible)* AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FT, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KB, KG, KM, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM ZW

(84) Países designados *(a menos que se indique lo contrario para cada clase de protección disponible)* ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasiática (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), Europea (AT, BE,

[Continúa en la página siguiente]

(54) Título: PROCESO Y APARATO DE SECADO



(57) Extracto: Esta invencion se refiere a un proceso y/o método y aparato asociado para uso en secar por congelamiento por aspersión una sustancia líquida, tal como jugo de fruta, productos farmacéuticos, nutracéuticos, té y café Una sustancia líquida se rocía (atomiza) y reduce en temperatura a por debajo de su temperatura eutectica, induciendo en esa forma un cambio de fase de líquidos en la sustancia líquida La sustancia líquida atomizada (ALS) se somete entonces a una cámara de secado de vacío, en la cual la ALS se somete a una fuente de energía para ayudar a la sublimación de líquido de la ALS, a medida que se conduce a través de la cámara Tal fuente de energía puede proporcionar una gradiente de temperatura, a través de la cual pasa la ALS y se expone a ella, todo llevado a cabo de modo que, preferiblemente, se minimice o reduzca la probabilidad de degradación de la sustancia que se está secando (reduciendo el contenido de líquido)

WO 2005/105253 A1

WO 2005/105253 A1

BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)

Para códigos de dos letras y otras abreviaturas, remítase a las "Notas Guía sobre Códigos y Abreviaturas" que aparece al comienzo de cada edición regular del Boletín Oficial PCT

Publicado:
— *con informe internacional de búsqueda*

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PROCESO Y APARATO DE SECADO

CAMPO TÉCNICO

Esta invención se refiere a un secador y a un proceso de secado. En particular, aunque no exclusivamente, esta invención se refiere a un secador para uso en congelar, concentrar o secar una sustancia líquida que tiene partículas sólidas en suspensión, o una sustancia disuelta en ella.

ARTE PREVIO

El proceso de la conservación de alimentos siempre ha sido de interés, siendo el secado por congelamiento el foco particular de la industria alimenticia desde finales de la década de los años 40. En los años de formación de los aparatos de secado por congelamiento y desarrollo de procesamientos, hubo un marcado acento en lograr un producto final útil (conservado). Las fases básicas de congelar un material líquido y, luego, calentar el material congelado bajo vacío para efectuar la extracción de la humedad son preferibles a las tentativas anteriores en la conservación de alimentos que, con frecuencia, involucraban deshidratación por métodos de convección de aire caliente.

La calidad del producto desecado es de importancia primordial en términos de valor nutricional y apariencia para el consumidor. No es deseable desmejorar el producto durante el procesamiento y extracción de líquidos de las sustancias. El secado por convección de aire caliente resulta a menudo en el encogimiento del producto y otros efectos perjudiciales.

Se han utilizado operaciones de unidades por lotes en la industria de secado por congelamiento, aunque el procesamiento por lotes tiene un número de desventajas que incluyen: períodos más prolongados del procesamiento del producto, tiempos de residencia más prolongados en cada fase de procesamiento (en muchos casos, ya que se requiere trabajo manual para trasladar sustancias parcialmente procesadas de una fase a la siguiente), equipo de procesamiento deficientemente mejorado, a menudo con exceso de capacidad, mayores tiempos de instalación, control reducido del proceso debido a la mayor probabilidad de error humano (como consecuencia de la falta de automatización), y rendimiento más bajo. Típicamente, los sistemas por lotes se emplean para ciclos pequeños de producción o donde se precisa flexibilidad del proceso.

En procesos convencionales de secado por congelamiento (en lotes), por lo menos un suministro líquido se vierte en bandeja(s) poco profunda(s) (el espesor del producto varía por lo general entre 10 y 20mm), la cual(es) se pone(n) luego en anaqueles en el secador por congelamiento. La puerta de acceso al congelador por lotes se cierra y el producto se congela. Luego de congelado el producto, las bandejas se calientan y el hielo se sublima lentamente. El vapor sublimado se condensa en serpentines refrigerados. Una vez se asume que el producto está seco, éste se extrae manualmente. El producto sale de un secador por congelamiento convencional en la forma de una masa compacta quebradiza y, usualmente, requiere una fase de granulación separada, antes de poderse procesar posteriormente.

Este método de secar una sustancia con carga de humedad congelándola, sublimando luego el exceso de humedad tanto como sea posible para generar un producto desecado, se utiliza principalmente en industrias donde se necesita secar sustancias incapaces de resistir temperaturas incluso moderadas; por ejemplo algunos productos alimenticios o farmacéuticos pueden estropearse o afectarse por el calor.

No es inusual que este proceso combinado de congelamiento y secado tome 48 horas o más. Esto no es deseable si se están secando materiales sensibles al calor. Además, si se pierde el vacío en un secador por congelamiento convencional por lotes, puede ocurrir descongelamiento y formación de cristales, y es posible que pueda perderse toda la carga del producto, es decir, el producto puede no ser un producto de calidad aprovechable, o no estar en capacidad de venderse; adicionalmente, el proceso de carga y descarga es susceptible a contaminación del producto, debido a la exposición, y a desperdicio por derrame de las bandejas poco profundas. Por lo tanto, es deseable un sistema que posibilite un rápido secado por congelamiento de una sustancia cargada de humedad y que genere un producto final convenientemente formado, tal como un polvo.

Hasta el momento, la industria de secado no ha desarrollado efectivamente procesos de secado por congelamiento verdaderamente continuos o semi-continuos.

Un aparato capaz de minimizar la contaminación potencial, reducir el derrame del suministro de líquido y que pueda aumentar el rendimiento líquido (*"the liquid throughput"*) sería ventajoso para la industria de secado. Si un suministro líquido es susceptible de secarse por congelamiento, sin requerir al mismo tiempo un pretratamiento necesario, fases de enfriamiento de un suministro líquido, o fases subsiguientes de granulación del producto seco, entonces pueden superarse problemas significativos con el secado por congelamiento convencional por lotes.

En tiempos más recientes, se han desarrollado aún más las fases de congelamiento y secado para incluir varios pasos, tales como reducir el tamaño físico de las sustancias a tratar, así como el control de temperatura graduado durante las fases de congelamiento y secado. No obstante, en la práctica, a menudo ha sido difícil implementar efectivamente un secado altamente controlado, ya que las sustancias que se están desecando se encuentran con frecuencia en arreglos de bandeja tipo estático y, en consecuencia, alguna parte de la sustancia tiende a verse más afectada o calentada que otras partes de la sustancia, con el resultado, por lo tanto, de un producto seco o calentado en exceso y, por consiguiente, estropeado. Por ello, sería ventajoso utilizar una fase de secado con la provisión de condiciones mejoradas de transferencia de calor a las sustancias, estado tal fase preferiblemente acoplada a un proceso de secado por congelamiento.

Por consiguiente, es un objeto de la presente invención proveer un aparato secador y/o un proceso de secado o método, que avance por lo menos un trecho en abordar los problemas anteriores o en proporcionar por lo menos una opción útil a la industria.

Todas las referencias, inclusive cualesquiera patentes o solicitudes de patente citadas en esta especificación se incorporan aquí por referencia. No se admite que cualquier referencia constituya arte previo. La discusión de las referencias establece que sus autores sostienen, y los solicitantes se reservan el derecho de cuestionar la exactitud y pertinencia de los documentos citados. Se entenderá claramente que, aunque aquí se hace referencia a un número de publicaciones del arte previo, esta referencia no constituye una admisión de que cualquiera de estos documentos forme parte del conocimiento general común en el arte, en Nueva Zelandia o en cualquier otro país.

Se acepta que, bajo jurisdicciones variables, al término 'comprender' puede atribuirse un significado inclusivo o exclusivo. Para el propósito de esta especificación y, a menos que se anote lo contrario, el término 'comprender' tendrá un significado inclusivo – es decir, que se tomará para significar una inclusión de no sólo los componentes enumerados a los que ésta hace directamente referencia, sino también otros componentes o elementos no especificados. Este razonamiento se usará igualmente cuando se use el término 'comprendido' o 'que comprende' en relación con uno o más pasos en un método o proceso.

Otros aspectos y ventajas de la presente invención se harán evidentes a partir de la siguiente descripción, la cual se da a manera de ejemplo únicamente.

DIVULGACIÓN DE LA INVENCIÓN

En consecuencia, en un primer aspecto, en sentido amplio la invención consta de un método de secar o concentrar una sustancia líquida, que tiene partículas sólidas en suspensión o una sustancia disuelta en ella, el cual comprende los pasos de:

mantener una cámara a una temperatura y presión por debajo del punto triple de la sustancia líquida,

inyectar y/o atomizar la sustancia líquida en la cámara para generar una porción de sustancia líquida congelada ("porción FLS" – ***frozen liquid substance***) y una primera porción de sustancia líquida evaporada ("porción FEL" – ***first evaporated liquid***),

recoger la porción FLS como una capa en una superficie, y

conducir la porción FLS recogida en la superficie,

en donde la superficie conduce la porción FLS recogida a una velocidad, que controla el espesor de la capa de FLS recogida en la superficie.

Una "sustancia líquida" se usa aquí y se refiere a una sustancia que tiene propiedades de flujo similares a un líquido (*"liquid flow-like properties"*), pero posee peso/masa constante en un primer estado. El peso/masa de la sustancia líquida puede alterarse extrayendo componentes de la sustancia líquida, por ejemplo evaporando líquidos de la sustancia, dejando una sustancia líquida más concentrada. Tales sustancias líquidas pueden incluir líquidos con partículas o sólidos suspendidos o disueltos en una solución, así como compuestos, que pueden existir en un estado líquido, tales como agua o aceite o similares.

Preferiblemente, la velocidad de conducción logra que sustancialmente un espesor de monocapa de la porción FLS se acumule en dicha superficie.

Preferiblemente, la FEL se condensa por un dispositivo condensador. Preferiblemente, la FEL se extrae de dicha cámara.

Preferiblemente, inyectar y/o atomizar la sustancia líquida incluye atomización para lograr un tamaño de partícula predeterminado de FLS.

Preferiblemente, la inyección y/o atomización de la sustancia líquida se realiza por una o más boquillas.

Preferiblemente, la velocidad de conducción de FLS sobre dicha superficie alcanza sustancialmente un espesor de monocapa de la FLS sobre dicha superficie.

Preferiblemente, la monocapa puede tener el espesor de una sola capa del tamaño de partícula predeterminado de la porción FLS.

Preferiblemente, el tamaño de partícula predeterminado es sustancialmente 500 µm o más.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 500 µm.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 200 µm.

Preferiblemente, la superficie conduce la porción FLS alejándola de una o más boquillas.

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Preferiblemente, el método incluye el paso de exponer la capa recogida de la porción FLS a un medio de calefacción, induciendo así sustancialmente la sublimación y generación de una segunda porción de sustancia líquida ("porción SEL" – ***second evaporated liquid***) y un producto.

Preferiblemente, la porción SEL se condensa por un dispositivo condensador.

Preferiblemente, la porción SEL se extrae de la cámara.

Preferiblemente, el producto se extrae de la superficie de conducción por un dispositivo de extracción del producto.

Preferiblemente, el producto se extrae de la cámara a través del puerto de salida.

Preferiblemente, el método se usa para procesar una sustancia fluidificada, o una sustancia líquida con partículas sólidas en suspensión, o una sustancia disuelta en ella.

Preferiblemente, la sustancia se selecciona de una o más de las siguientes: una suspensión de café, leche líquida, jugos de frutas y/o vegetales.

En un segundo aspecto, en sentido amplio la invención consta de un aparato para secar o concentrar una sustancia líquida, el cual comprende:

una cámara y un dispositivo de reducción de presión de la cámara, y

uno o más puertos de inyección, a través de los cuales la sustancia líquida se inyecta a la cámara,

una superficie de recolección que recoge una porción de sustancia líquida congelada de la sustancia líquida,

en donde el dispositivo de reducción de presión mantiene la cámara a una presión por debajo de por lo menos la presión de punto triple de la sustancia líquida, para hacer que la sustancia líquida inyectada se separe en una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de sustancia líquida evaporada ("porción FEL"),

de modo que, en uso, la porción FLS se acumule como una capa sobre la superficie de recolección y se conduzca alejándose del uno o más puertos de inyección a una velocidad que permita controlar el espesor de la capa de la porción FLS.

Preferiblemente, la velocidad de conducción logra que sustancialmente un espesor de monocapa de la porción FLS se acumule en dicha superficie.

Preferiblemente, la porción FEL se condensa por un dispositivo condensador.

Preferiblemente, dicha FEL se extrae de dicha cámara.

Preferiblemente, los uno o más puertos de inyección comprenden por lo menos una boquilla de aspersión o atomización.

Preferiblemente, la boquilla o boquillas determinan sustancialmente el tamaño de la porción FLS generada para alcanzar un tamaño de partícula predeterminado.

Preferiblemente, el dispositivo de reducción de presión es una bomba de evacuación de gas.

Preferiblemente, el dispositivo condensador es un serpentín o serpentines refrigerados.

Preferiblemente, el dispositivo condensador se enfría con un refrigerante.

Preferiblemente, la superficie de recolección incluye uno, o una combinación de, los siguientes transportadores mecánicos: una configuración de cinta sinfín, una bandeja inclinada para favorecer que la porción de sustancia líquida congelada se deslice alejándose del uno o más puertos de inyección, una bandeja vibradora.

Preferiblemente, la bandeja inclinada y/o bandeja vibradora incluye una superficie de fricción reducida.

Preferiblemente, la superficie de fricción reducida comprende politetrafluroetileno (PTFE).

Preferiblemente, la capa de la porción FLS sobre dicha superficie de recolección se expone a un medio de calefacción, a medida que se conduce alejándose del uno o más puertos de inyección.

Preferiblemente, el medio de calefacción induce sustancialmente sublimación de la capa de la porción FLS sobre dicha superficie para formar una segunda porción de sustancia líquida evaporada ("SEL" – ***second evaporated liquid***) y un producto.

Preferiblemente, el medio de calefacción es una, o una combinación, de las siguientes fuentes de energía: lámparas infrarrojas, lámparas halógenas, lámparas incandescentes, microondas o calentamiento óhmico de dicha superficie.

Preferiblemente, se emplea un dispositivo de extracción del producto para extraer el producto de la superficie de recolección.

Preferiblemente, el dispositivo de extracción del producto es un medio raspador y/o un medio de arrastre de cepillo. Preferiblemente, el medio raspador contacta sustancialmente la superficie, con el producto sobre ella, y traslada el producto desde la superficie al puerto de salida.

Preferiblemente, el medio de arrastre de cepillo es un cepillo giratorio o un cepillo fijo, que sustancialmente contacta la superficie, con el producto sobre ella, y dirige el producto de la superficie al puerto de salida.

Preferiblemente, el producto se extrae a través de un puerto de salida de la cámara. Preferiblemente, la cámara se mantiene a una presión de sustancialmente 611.3 Pa o menos.

En un tercer aspecto, en sentido amplio la invención consta de un proceso de secado para una sustancia líquida, el cual comprende los pasos de:

atomizar la sustancia líquida,

enfriar la sustancia líquida atomizada (ALS – ***atomised liquid substance***) para iniciar un cambio de fase,

conducir la ALS a una cámara de secado bajo vacío,

calentar la ALS con el fin de efectuar sustancialmente sublimación y, luego, recolectar la ALS sustancialmente seca de la cámara.

Preferiblemente, la sustancia líquida se enfría desde una temperatura inicial antes de atomizarla.

Preferiblemente, la ALS conducida a la cámara de secado se conduce también a través de la cámara de secado.

Preferiblemente, la ALS se calienta mediante una gradiente de temperatura mientras se desplaza a través de la cámara de secado, de modo que la gradiente de temperatura efectúa sustancialmente sublimación.

Preferiblemente, el paso de enfriar una sustancia líquida tiene lugar en un

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enfriador para reducir la sustancia líquida desde una temperatura inicial a una temperatura más baja.

Preferiblemente, atomizar la sustancia líquida alcanza un tamaño de partícula predeterminado.

Preferiblemente, el tamaño de partícula predeterminado es sustancialmente 500 μm o más.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 500 μm.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 200 μm.

Preferiblemente, un congelador por atomización utiliza un gas frío para efectuar un cambio de fase de los líquidos en la sustancia líquida atomizada (ALS).

Preferiblemente, el enfriamiento se lleva a cabo con contacto directo o indirecto con un fluido frío.

Preferiblemente, dicho fluido frío es aire.

Preferiblemente, dicho fluido frío está sustancialmente a 0°C o menos.

Preferiblemente, dicho fluido frío está a menos de sustancialmente -20°C.

Preferiblemente, dicho congelador por atomización opera en una configuración contracorriente.

Preferiblemente, dicho congelador por atomización opera en una configuración de la misma corriente.

Preferiblemente, la ALS se reduce a una temperatura por debajo de su temperatura eutéctica.

Preferiblemente, la ALS se conduce a un separador.

Preferiblemente, dicha ALS se conduce neumáticamente a un separador.

Preferiblemente, dicho separador es un dispositivo de separación gas-sólido.

Preferiblemente, dicho dispositivo de separación gas-sólido es un ciclón.

Preferiblemente, el gas separado en el dispositivo de separación gas-sólido se realimenta y/o refrigera para uso en el paso de enfriamiento.

Preferiblemente, uno o más cierres de vacío y/o aire están presentes entre una salida del ciclón AFS y una entrada de la cámara de secado.

Preferiblemente, sólidos separados de dicho dispositivo de separación gas-sólido ingresan a una cámara de secado de vacío.
Preferiblemente, el vacío de dicha cámara de secado de vacío se crea por un dispositivo de reducción de presión.
Preferiblemente, el vacío creado por el dispositivo de reducción de presión es sustancialmente 600 micrómetros Hg de presión absoluta o menos.
Preferiblemente, el vacío creado por el dispositivo de reducción de presión está en el rango de sustancialmente 200-400 micrómetros Hg de presión absoluta.
Preferiblemente, dicha ALS se conduce mediante una superficie.
Preferiblemente, dicha ALS se conduce mediante una superficie vibradora.
Preferiblemente, dicha superficie vibradora es una bandeja vibradora.
Preferiblemente, dicha bandeja vibradora se acciona neumática y/o mecánica y/o eléctrica y/o magnéticamente.
Preferiblemente, dicha bandeja vibradora conduce la ALS a través de una gradiente de temperatura para efectuar sustancialmente sublimación.
Preferiblemente, un vapor producido por sublimación se extrae de la cámara de secado.
Preferiblemente, vapor producido por sublimación se extrae de la cámara de secado por uno o más condensadores.
Preferiblemente, la ALS se calienta mediante una gradiente de temperatura mientras se desplaza a través de la cámara de secado, de modo que la gradiente de temperatura efectúa sustancialmente sublimación.
Preferiblemente, dicha gradiente de temperatura se provee por una fuente de energía.
Preferiblemente, dicha fuente de energía es un dispositivo emisor infrarrojo y/o un dispositivo emisor microondas y/o un calentador óhmico.
Preferiblemente, atomizar la sustancia líquida alcanza un tamaño de partícula predeterminado.
Preferiblemente, el tamaño de partícula predeterminado es sustancialmente

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500 µm o más.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 500 µm.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 200 µm.

Preferiblemente, se emplea un sistema de refrigeración para mantener la ALS a una temperatura por debajo de su temperatura eutéctica durante el transporte desde el dispositivo de separación gas-sólido a la cámara de secado de vacío.

Preferiblemente, el producto generado sustancialmente reducido está libre de líquido, comparado con la sustancia líquida.

En un cuarto aspecto, en sentido amplio la invención consta de un aparato para un proceso de secado de una sustancia líquida, el cual comprende:

un atomizador capaz de atomizar una sustancia líquida,

un enfriador capaz de enfriar la sustancia líquida atomizada (ALS) para iniciar un cambio de fase,

un transportador mecánico (*"a conveyor"*) capaz de conducir la ALS a una cámara de secado mantenida bajo vacío,

una fuente de energía capaz de calentar la ALS con el fin de efectuar sublimación y secado por congelamiento, y

un recolector capaz de recolectar la ALS seca.

Preferiblemente, antes del atomizador de sustancia líquida se provee un enfriador, capaz de enfriar una sustancia líquida desde una temperatura inicial a una temperatura más baja.

Preferiblemente, el aparato incluye un transportador mecánico capaz de conducir la ALS a través de la cámara de secado.

Preferiblemente, el aparato incluye una fuente de energía capaz de calentar la ALS mediante una gradiente de temperatura, mientras se desplaza a través de la cámara de secado, de manera que la gradiente de temperatura pueda efectuar sublimación.

Preferiblemente, la gradiente de temperatura sustancialmente previene o sustancialmente minimiza el deterioro por calor que ocurre a la ALS a

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medida que ésta pasa a través de la cámara.

Preferiblemente, atomizar la sustancia líquida alcanza un tamaño de partícula predeterminado.

Preferiblemente, el tamaño de partícula predeterminado es sustancialmente 500 µm o más.

Preferiblemente, el tamaño de partícula predeterminado es menos de sustancialmente 500 µm.

Preferiblemente, el tamaño predeterminado es menos de sustancialmente 200 µm.

Preferiblemente, un congelador por atomización utiliza un gas frío para efectuar un cambio de fase de los líquidos en la sustancia líquida atomizada (ALS).

Preferiblemente, dicho gas frío es aire.

Preferiblemente, dicho gas frío está sustancialmente a 0°C o menos.

Preferiblemente, dicho gas frío está a menos de sustancialmente -20°C.

Preferiblemente, el vacío de dicha cámara de secado de vacío se crea por un dispositivo de reducción de presión.

Preferiblemente, el vacío creado por el dispositivo de reducción de presión es sustancialmente 600 micrómetros Hg de presión absoluta o menos.

Preferiblemente, el vacío creado por el dispositivo de reducción de presión está en el rango de sustancialmente 200-400 micrómetros Hg de presión absoluta.

Preferiblemente, dicha ALS se conduce mediante una superficie vibradora.

Preferiblemente, dicha superficie vibradora es una bandeja vibradora.

Preferiblemente, dicha bandeja vibradora se acciona neumática y/o mecánica y/o eléctrica y/o magnéticamente.

Preferiblemente, la ALS se conduce a través de una gradiente de temperatura para efectuar sustancialmente sublimación.

Preferiblemente, el vapor producido por sublimación se extrae de la cámara de secado por uno o más condensadores.

Preferiblemente, dicha gradiente de temperatura se provee por una fuente de energía.

Preferiblemente, dicha fuente de energía es un dispositivo emisor infrarrojo y/o un dispositivo emisor microondas y/o un calentador óhmico.

En un quinto aspecto, en sentido amplio la invención consta de una cámara de secado que comprende:

un dispositivo capaz de hacer vibrar una bandeja,

un dispositivo apropiado para reducir la presión en dicha cámara,

un puerto de salida de material de dicha cámara, y

una fuente de calor adaptada para actuar sobre dicha bandeja.

Ventajosamente, la presente invención provee un método/proceso mejorado de tratar una sustancia líquida para fabricar una forma más seca o por lo menos más concentrada de la sustancia líquida (en la forma de un producto).

En incorporaciones preferidas, la presente invención está en capacidad de ser operada sobre una base semi-continua, o que por lo menos permitirá un mayor rendimiento de sustancia líquida, comparada con sistemas de secado del arte previo. La velocidad de conducción de la sustancia líquida atomizada (ALS) es suficiente para incrementar las eficiencias de secar/concentrar y/o que contribuye a reducir el deterioro al producto.

BREVE DESCRIPCIÓN DE LOS DIBUJOS

Otros aspectos de la presente invención se harán evidentes a partir de la siguiente descripción, dada a manera de ejemplo únicamente, y con referencia a los dibujos adjuntos en los que:

Figura 1 ilustra una incorporación de una posible configuración de un secador, de acuerdo con la presente invención en los aspectos tercero y cuarto.

Figura 2 Es un diagrama de flujo de un proceso de una posible incorporación de la presente invención, de acuerdo con los aspectos primero y segundo;

Figura 3 es una vista superior de la configuración de la cámara de secado mostrada en Figura 2,

Figura 4 es una vista lateral de la configuración de la cámara de secado mostrada en Figuras 2 y 3, y

Figura 5 es una vista final de la configuración de la cámara de secado mostrada en Figuras 2 a 4.

MEJORES MODOS DE LLEVAR A CABO LA INVENCIÓN

La presente invención puede describirse ahora con referencia a Figuras 1 a 5, y la descripción que la acompaña.

La presente invención se refiere a un proceso para secar por congelamiento o deshidratar/concentrar sustancias líquidas. El proceso es aplicable a líquidos que tienen una sustancia sólida en suspensión (por ejemplo, leche), o a soluciones líquidas en las cuales se ha disuelto una sustancia. Usando este secador por congelamiento por atomización podrían procesarse igualmente té, café, jugo de fruta, productos farmacéuticos y nutracéuticos.

El secado por congelamiento es una útil técnica de conservación y, entre muchos otros productos, los siguientes pueden secarse de esta manera. café instantáneo, vegetales para mezclas de sopas deshidratadas, champiñones, hierbas aromáticas, condimentos, cultivos de microorganismos para la fabricación de queso (*"cheese starter cultures"*), camarones, frutas de cereales de desayunos listos para comer, nutracéuticos, productos farmacéuticos y agricéuticos.

Algunos usuarios finales específicos de productos secados por congelamiento pueden incluir la producción de raciones militares y/o espaciales, así como alimentos de peso liviano con contenido de vegetales, carne, pescado y frutas, consumidos por campistas. Por lo general, el proceso de secado por congelamiento puede tener las siguientes ventajas: bajo deterioro térmico, excelente retención de sabores volátiles, buena retención de vitaminas, rápida rehidratación del producto, bajo encogimiento del producto, vida útil prolongada del producto – si se empaca adecuadamente - y retención de la actividad biológica. Aunque hay también algunas desventajas asociadas con secar por congelamiento sustancias, contándose entre las más notables: costo elevado de secado, daño infligido a ciertos productos por el congelamiento inicial, descomposición rápida, a menos que se empaquen y mantengan con baja humedad, friabilidad (es decir, se desmoronan fácilmente); a menudo, necesidad de pretratamiento (por ejemplo, zanahorias) para evitar la pérdida de color.

Un proceso **1**, como se describe generalmente en el tercer aspecto, y un aparato, como se define ampliamente por el cuarto aspecto ya descrito, comprende mantener una cámara **2** a una temperatura y presión por debajo del punto triple de una sustancia líquida **3**, inyectar la sustancia líquida **3** en la cámara **2** generando en esa forma una porción de sustancia líquida congelada **4** y una primera porción de sustancia líquida evaporada **5A**.

La primera porción de la sustancia líquida evaporada **5A** puede condensarse por el medio condensador **6**, mientras se recoge al mismo tiempo la porción de sustancia líquida congelada **4**, como una capa **7**, en una superficie de recolección **8**.

La porción de sustancia líquida congelada **4** recogida como la capa **7** se conduce a lo largo de la superficie de recolección **8**. La superficie **8** conduce la capa de la porción de sustancia líquida congelada **7** a una velocidad que controla el espesor de la capa de la porción de sustancia líquida congelada recogida.

La primera porción de la sustancia líquida evaporada condensada puede extraerse de la cámara **2**.

Con el fin de eliminar cualquier volumen de agua o porción de sustancia líquida evaporada de la fase gaseosa en la cámara, aquella puede condensarse en los serpentines. Con el fin de eliminar la porción evaporada de los serpentines condensadores, los serpentines pueden retornarse a presiones por encima del punto triple, de modo que el hielo se derrita. Por lo tanto, con el fin de obtener una operación sustancialmente continua o cuasi-continua, pueden utilizarse dos o más juegos de serpentines de condensación; mientras uno está formando hielo (*"is frosting up"*), el otro o los serpentines restantes pueden aislarse de la cámara de secado cerrando un sello de presión de algún tipo, retornando a temperatura ambiente, y descongelarlos.

La sustancia líquida **3** en un tanque de conservación **T** puede atomizarse en la cámara **2** con el fin de conseguir un tamaño predeterminado de partículas de la porción de sustancia líquida congelada, y esto puede realizarse por una o más boquillas **10**.

El espesor de la porción de sustancia líquida congelada (partículas) recogida en la superficie de recolección se determina por la velocidad (metros por segundo) de conducción por la superficie de recolección **8**, que es en forma de un transportador mecánico sinfín (no mostrado). El espesor de esta monocapa de partículas se determinará asimismo por los tamaños de partícula generados por la inyección de la sustancia líquida en la cámara y/o de la inyección de sustancia líquida por la boquilla(s) **10**.

Deseablemente la superficie conduce la porción de sustancia líquida congelada alejándola del punto de inyección de sustancia líquida, o la posición de una o más boquillas, a una velocidad que sustancialmente alcanza aproximadamente el espesor de monocapa de la porción de sustancia líquida congelada sobre la superficie de recolección **8**.

La porción de sustancia líquida congelada recogida puede exponerse entonces a un medio de calefacción **11**, el cual puede inducir por lo menos alguna sublimación y, con ello, generación de una segunda porción de sustancia líquida evaporada **5B** y un producto **12**.

La segunda porción de sustancia líquida evaporada **5B** puede condensarse asimismo por el medio condensador **6** y extraerse de la cámara **2**.

El producto **12** (en un estado que, ventajosamente, contiene menos líquido que en el estado en tanque de conservación **T**) puede extraerse de la superficie de recolección **8** por un medio de extracción del producto **13**, luego evacuarse de la cámara **2** a través del puerto de salida **14**.

En una incorporación adicional del proceso, sustancialmente como ya se describió, puede configurarse un aparato para generar un producto **12**, que es más seco o más concentrado que el de la sustancia líquida **3** en el tanque de conservación **T**.

Una cámara **2** se mantiene a una presión, por lo menos por debajo de la presión de punto triple de la sustancia líquida, por un medio de reducción de presión, tal como una bomba de evacuación de gas **15**.

Pueden emplearse uno o más puertos de inyección, tal como boquillas **10**, a través de las cuales puede atomizarse la sustancia líquida **3**. Luego de la inyección (o atomización) de la sustancia líquida **3** en la cámara **2**, pueden generarse una porción de sustancia líquida congelada **4** y una primera porción de la sustancia líquida evaporada **5A**.

Una superficie de recolección **8** se provee sustancialmente para recoger la porción de sustancia líquida congelada generada **4** de la sustancia líquida **3**. Puede emplearse un medio condensador **6** para condensar la primera porción de la sustancia líquida evaporada **5A**, y el condensado puede drenarse posteriormente y extraerse de la cámara **2**.

Un puerto de salida **14** de la cámara puede usarse para evacuar una forma más seca o más concentrada de la sustancia líquida **3**, en la forma de un producto **12**.

La porción de sustancia líquida congelada **4** puede acumularse como una capa **7** en la superficie de recolección y conducirse desde el uno o más puertos de inyección hacia el puerto de salida, a una velocidad que permite la acumulación de no más de sustancialmente un espesor de monocapa de la porción de sustancia líquida congelada.

La primera porción de la sustancia líquida evaporada **5A** puede condensarse por el medio condensador **6** y extraerse de la cámara. El medio condensador **6** es un serpentín o serpentines de condensación refrigerados. El medio condensador se refrigera con un refrigerante, suministrado por la unidad de refrigeración **16**.

El uno o más puertos de inyección comprenden una boquilla o boquillas de aspersión o atomización. La boquilla o boquillas determinan sustancialmente el tamaño de la porción de sustancia líquida congelada generada.

La superficie de recolección incluye uno, o una combinación, de los siguientes transportadores mecánicos: una configuración de cinta sinfín, una bandeja inclinada para favorecer que la porción de sustancia líquida congelada se deslice del uno o más puertos de inyección, una bandeja vibradora. La bandeja inclinada y/o bandeja vibradora incluye una superficie de fricción reducida, que comprende politetrafluroetileno.

La capa de porción de sustancia líquida congelada sobre dicha superficie de recolección se expone a un medio de calefacción, a medida que se conduce alejándola del uno o más puertos de inyección, lo cual puede sustancialmente inducir sublimación de la capa de porción de sustancia líquida congelada **7** sobre la superficie **8** para formar una segunda porción de sustancia líquida evaporada **5B** y un producto **12**.

El medio de calefacción es una, o una combinación, de las siguientes fuentes de energía: lámparas infrarrojas, lámparas halógenas, lámparas incandescentes, microondas o calentamiento óhmico de la superficie **8** misma, el cual puede actuar directamente sobre la porción de sustancia líquida congelada **4**. El medio de calefacción puede operar también indirectamente sobre las partículas congeladas mediante transferencia de calor proveniente del equipo de cámara interna circundante, tal como transferencia de calor a través de la superficie de recolección **8**, o mediante radiación o reflexión de energía proveniente de las paredes de la cámara circundantes.

Puede emplearse un dispositivo de extracción del producto para extraer el producto de la superficie de recolección, que puede haberse pegado o adherido a la superficie. Tal dispositivo de extracción del producto puede ser un medio raspador y/o un medio de arrastre de cepillo.

Un medio raspador puede contactar sustancialmente la superficie **8**, con el producto **12** sobre ella, y mover el producto de la superficie hacia el puerto de salida **14**, mientras que un medio de arrastre de cepillo puede estar en la forma de un cepillo giratorio o cepillo fijo, que sustancialmente contacta también la superficie, con el producto sobre ella, y desplaza el producto hacia el puerto de salida. Una vez extraído el producto de la cámara, se puede entonces empacar o procesar adicionalmente (tal como una fase de granulación, o empacado por vacío, para mantener la calidad del producto).

La sustancia líquida **3** puede suministrarse bajo presión al punto de inyección **10** mediante una bomba **17**, pasando la sustancia líquida al ambiente despresurizado de la cámara **2** a través de una serie de válvulas

18A.

La porción congelada **5A** de la sustancia líquida inyectada rociada o atomizada **3** se dirige ventajosamente para posarse y recogerse sobre una superficie **8**, la cual conduce entonces la porción congelada **4** alejándola de la boquilla(s) de inyección **10**.

La superficie **8** facilita el desplazamiento de la porción congelada **4** lejos de la boquilla(s) de inyección mediante el uso de una, o una combinación, de opciones de superficie de transportador mecánico. Se proveen estas opciones de superficie, las cuales permiten que la porción congelada recogida en la superficie **4** preferiblemente no se acumule en una proporción mayor a aproximadamente un espesor de monocapa de porción congelada **4**. El espesor o capa **7** de la porción congelada **4** sobre la superficie **8** determina directamente la velocidad de (secado) durante la exposición al medio de calefacción **11**.

Alternativamente, un espesor óptimo para sublimación rápida (secado) de la porción congelada puede determinarse midiendo activamente el espesor de la capa en la superficie, por ejemplo usando técnicas de reflexión de luz. Una vez determinado el espesor de la capa que se está atomizando sobre la superficie, la velocidad del transportador mecánico para alejar las partículas del puerto(s) de inyección puede alterarse para que corresponda a la velocidad de aspersión de entrada de la sustancia líquida, o para optimizar el rendimiento de la sustancia líquida (y la generación de producto). La velocidad de aspersión o rendimiento de sustancia líquida puede alterarse asimismo para que sea compatible con la velocidad de conducción de las partículas a lo largo de la superficie.

Una vez expuesta la porción congelada **4** al medio de calefacción **11**, la sublimación se induce preferiblemente de ese modo, lo cual resulta en una extracción de líquido adicional de la sustancia líquida ahoya congelada. La sustancia con contenido de humedad sustancialmente reducido, que puede designarse como el 'producto' **12**, puede extraerse entonces de la superficie de conducción. El producto puede recogerse luego y extraerse de la cámara **2** a través de un puerto de salida **14**, el cual incluye una o más válvulas **18B** (o cierres de aire) para evitar la pérdida de vacío de la cámara.

Como la cámara **2** se mantiene a un vacío, se requiere una bomba de vacío **15** para obtener las presiones deseadas en la cámara, por ejemplo, el punto triple del agua es 611.3 Pa (ó 4.585 mm Hg, ó 0.0887 psi). La presión de la cámara no tiene nunca que ser superior a la presión de punto triple del agua; no obstante, dependiendo del producto, tienen que cumplirse ciertas restricciones de temperatura para evitar la formación de cristales y la depresión del punto de congelamiento, asociada con los efectos de concentración de congelamiento, que ocurren a medida que el producto se seca. Algunos productos tienen que mantenerse por debajo de su temperatura eutéctica, mientras que para otros la temperatura de transición vítrea es crítica. Puesto que la presión en el recipiente determinará la temperatura de sublimación y, por consiguiente, la temperatura del producto, a menudo el proceso tendrá que operar a presiones significativamente por debajo de 611.3 Pa, por ejemplo, café: -21°C y 94 Pa; jugos de frutas: -30°C y 38 Pa. Naturalmente, se reconoce que esta tecnología puede aplicarse a la mayoría de las sustancias líquidas, que contienen partículas en suspensión o un material disuelto en ellas. Una persona experta en el arte de la termodinámica entendería la relación de presión y temperatura, y cómo las condiciones de la cámara determinan la cantidad de evaporación instantánea (generación de la primera porción de la sustancia líquida evaporada) y temperatura resultante de la porción de sustancia líquida congelada formada. Las condiciones de la cámara pueden variarse para optimizar el nivel de rendimiento y/o nivel de congelamiento que ocurren en la cámara.

Como el suministro de líquido puede alimentarse a través de una boquilla directamente a la cámara de vacío, la boquilla y la presión de alimentación se seleccionan de tal manera que se mantenga un chorro de atomización estable. En un ambiente por debajo del punto triple (*"a sub-triple point environment"*), un material no puede existir en el estado líquido, lo que significa que cierta porción del líquido es forzada a vaporizarse casi instantáneamente, haciendo que el líquido restante se congele. Puesto que el trabajo de entalpía de fusión no necesita suministrarse por una fuente de energía externa, es efectivamente "libre". Adicionalmente, como resultado del mismo proceso, aproximadamente un séptimo del trabajo de sublimación es también libre. Esto significa que para una carga dada de producto, el secador por congelamiento por atomización requerirá por lo menos 10% menos de energía que un secador por congelamiento convencional por lotes.

El producto se congela en partículas finísimas, cuyo tamaño máximo se limita por la tensión superficial y la presión de vapor (típicamente 100μm o menos). Las partículas congeladas se posan en una bandeja calentada o cinta transportadora, donde se secan a medida que la humedad se sublima y se condensan por los serpentines condensadores, a medida que se conducen a todo lo largo del secador. Los tiempos de liofilización pueden calcularse a partir de la siguiente ecuación (Fellows, 1997):

$$t_d = \frac{x^2 \rho \Delta M \lambda_s}{8 k_d \Delta T}$$

Donde. t_d = tiempo(s) de secado, x = espesor de la capa de producto (m), k_d = conductividad térmica del producto seco (W/mK), ρ = densidad aparente del producto (kg/m^3), ΔM = variación en el contenido de humedad (base seca), ΔT = fuerza de transferencia de temperatura (°C) y λ_s = entalpía de sublimación (J/kg). Esta ecuación muestra que el tiempo de secado es proporcional al cuadrado del espesor de la capa de producto sobre la superficie de secado.

La presente invención del segundo aspecto puede designarse como un proceso de secado por congelamiento por atomización en un proceso cuasi-continuo, y puede integrarse en una línea de producción continua, que reduciría los requerimientos de mano de obra asociados con su operación, y podría eliminar asimismo el riesgo de exposición o contaminación del producto. Adicionalmente, se elimina la necesidad de una fase adicional de granulación, puesto que el producto seco sale del secador en forma de polvo.

Una superficie recubierta con politetrafluroetileno (PTFE), tal como un sistema de conducción de cinta transportadora, puede acoplarse a un motor de velocidad variable, que permita variar el tiempo de residencia del producto sobre la cinta transportadora entre aproximadamente 1 y 12 minutos, dependiendo de la exposición al medio de calefacción, requerida para sublimar una cantidad deseada de humedad. La longitud de la cinta transportadora puede ser de alrededor de 2m, y las velocidades de la cinta transportadora pueden variarse entre alrededor de 0.17m/min y 2m/min. El producto puede atomizarse a través de boquillas múltiples, por ejemplo 4 boquillas en paralelo, con el fin de distribuir el producto uniformemente a lo ancho de la cinta transportadora (que puede ser aproximadamente 0.5m). Se preve que la mayoría de los tamaños de partícula estarán en la región de 10 a 100μm y, por consiguiente, puesto que es probable que el producto se adhiera a la superficie (o cinta transportadora) incluso con la cubierta de PTFE (politetrafluroetileno), un medio de extracción tal como el filo de una cuchilla, o cepillo o arreglo de medios raspadores puede emplearse al final del transportador mecánico (o cualquier posición a lo largo de la superficie). Si el raspador o filo de cuchilla no es lo suficientemente efectivo, puede usarse igualmente PTFE u otro tipo de material del cepillo para raspar las partículas. El arreglo de cepillo puede producir la agregación de las partículas, lo cual puede ser o no benéfico, dependiendo del manejo corriente abajo, previsto para el producto particular.

La boquilla de alimentación **10** puede sobresalir directamente en la cámara de vacío **2**. La presión dentro de cámara de vacío **2** se mantiene por debajo de la presión de punto triple del líquido **3** y, por lo tanto, a medida que la sustancia líquida **3** sale de la boquilla **10** alguna parte del agua se evapora **5A** instantáneamente ("se evapora rápidamente"), como resultado de la caída de presión por debajo de la presión de punto triple del líquido. El proceso de evaporación elimina cantidades sustanciales de calor de la goticula, con el resultado de que la humedad restante en el producto se congela. Basándose en las magnitudes relativas de los calores latentes de vaporización (= 2.250 kJ/kg) y fusión (≈ 333 kJ/kg), 1 gramo de humedad evaporada congelaría aproximadamente 6.5 gramos de agua residual **4**. La humedad restante en la porción de sustancia líquida congelada **4** puede eliminarse opcionalmente sometiéndola a un medio de calefacción o fuente de energía, tal como infrarroja, lámpara halógena u otra fuente de energía **11** en la cámara de vacío.

Ventajas potenciales de este tipo de arreglò incluirían además la significativa reducción en el requerimiento de energía externa (electricidad), ya que casi no se requiere electricidad para congelar el producto, así como la reducción asociada en el requerimiento de electricidad para calentar la bandeja; y pueden eliminarse en gran medida los problemas asociados con la conducción del producto congelado (prensado, compactación, aglomeración, descongelamiento/re-congelamiento).

El secador por congelamiento por atomización involucra menos equipo (y, por consiguiente, un menor costo de capital), debido a su diseño mecánicamente más sencillo, y existe el potencial de ejercer un control mucho mayor de la distribución lateral del producto en la bandeja de secado (porque el patrón de atomización puede controlarse mientras que, antes, el producto congelado simplemente se dejaba caer sobre la bandeja).

Algunas de las principales ventajas del secado por congelamiento por atomización sobre el secado por congelamiento convencional por lotes son los tiempos de secado altamente reducidos (una cuestión de segundos, comparada con una cuestión de horas); se minimiza la posibilidad de deterioro a materiales sensibles al calor; una reducción de aproximadamente el 10% en costos de energía debidos al novedoso proceso de congelamiento, que es efectivamente libre de costo, y se suprime la necesidad de una fase de granulación adicional, la cual es, a menudo, costosa y puede traducirse en pérdida de producto.

Utilizando este proceso cuasi-continuo, se evita la exposición a contaminantes y el secador por congelamiento por atomización es susceptible de integrarse en una línea de producción continua, y es mucho menos intensivo en mano de obra que los secadores por congelamiento convencionales.

En otros aspectos de la presente invención, como se define en sentido amplio por los aspectos primero, segundo y/o quinto, se provee un proceso y aparato de secado, como se ilustra por un diagrama de flujo del proceso continuo y mejorado de secado pro congelamiento por atomización, como se muestra Figuras 2, 3, 4 y 5. Una sustancia líquida, tal como café (no mostrada), se enfría desde su temperatura inicial a una temperatura un poco menor en un enfriador **21**. Este paso es opcional. El suministro de líquido se dirige bajo presión mediante una bomba (no mostrada), atomizado a través de una boquilla de alta presión (no mostrada) a una cámara de congelamiento **22**. Idealmente, el tamaño de partícula de la sustancia líquida atomizada debe ser menos de sustancialmente 200-300 micrones (μm). El suministro se introduce en la cámara de congelamiento **22** con un flujo de la misma corriente de aire frío (-20°C o menos) y se atomiza y congela para producir una sustancia congelada atomizada (ALS). La ALS se congela rápidamente por debajo de su temperatura eutéctica para evitar el derretimiento. La ALS congelada se conduce neumáticamente por la corriente de aire frío al congelador por atomización [y] a un ciclón separador **23**, donde la ALS congelada sale del fondo del ciclón **23** y el chorro de aire de salida sale de la parte superior del ciclón **23**. El chorro de aire de salida del congelador por aspersión tiene que estar por debajo de la temperatura eutéctica de la ALS congelada. Un ventilador o soplador **24** soplan el chorro de aire de salida proveniente del ciclón a través del sistema de refrigeración, en este caso un evaporador **25**, donde el aire se vuelve a enfriar a su temperatura inicial, antes de retornar a la cámara de congelamiento por atomización **22**.

El producto congelado cae luego por gravedad en una cámara de terminado (no mostrada), acoplada al fondo del ciclón. Un cierre de vacío o aire **27** permite que el producto pase sin interrupción, desde el fondo del ciclón a la cámara de secado **28**, al vacío dentro de la cámara de secado. La cámara de terminado y el cierre de aire se enfrían con aire frío proveniente del sistema de refrigeración, en este caso un enfriador **26** para evitar que la ALS se derrita. La cámara de secado se mantiene a una presión absoluta de sustancialmente 200-400 micrómetros de Hg con una bomba de vacío **212**. La ALS congelada cae sobre una bandeja vibradora neumática inclinada **29**, que conduce el producto seco hacia el extremo de descarga del secador. Para efectuar sublimación y secado por congelamiento, la bandeja se calienta por una fuente de calor radiante y/o de conducción **210**, ubicada debajo de la bandeja **29**. La temperatura de la superficie de la bandeja se controla a lo largo de su longitud para garantizar el secado completo sin deteriorar el producto (no mostrado). La ALS seca (producto) sale a través del cierre de vacío de descarga **213**. Se requieren dos condensadores **211** para operar el sistema en forma continua. Un condensador operaría normalmente durante el secado, mientras que el otro se aislaría de la cámara de secado y descongelaría.

Un proceso de secado mejorado para una sustancia líquida, de acuerdo con el primer aspecto, incluye uno o más de los pasos de enfriar la sustancia líquida desde una temperatura inicial, atomizar la sustancia líquida, de manera que se alcance un tamaño de partícula predeterminado, congelar la sustancia líquida atomizada (ALS) a por debajo de su temperatura eutéctica, conducir la ALS congelada a una cámara de secado que se mantiene bajo vacío, calentar la ALS congelada con el fin de efectuar sublimación y secado por congelamiento y, a continuación, recolectar la ALS seca de la cámara.

Una sustancia líquida puede definirse como cualquier sustancia que contiene un líquido. Éstas pueden incluir por ejemplo, leche, café, solución acuosa de compuestos químicos (*"liquor"*), productos farmacéuticos, nutracéuticos, alimento funcional, agricéuticos, o cualquier otra sustancia que tenga un contenido de humedad. La cantidad de líquido en una sustancia necesita ser tan sólo mínima para permitir, durante un período de tiempo, la degradación de la sustancia; por consiguiente, es deseable la eliminación de cualquier líquido "libre".

La conducción de ALS a una cámara de secado puede lograrse por cualesquiera métodos/aparatos apropiados de conducción. Por ejemplo, éstos pueden incluir métodos neumáticos, métodos mecánicos; también pueden ser apropiados métodos de conducción eléctricos o asistidos por gravedad. En otra incorporación del primero y/o segundo aspecto, preferiblemente se provee un proceso de secado mejorado para una sustancia líquida, el cual puede incluir uno o más de los pasos de enfriar la sustancia líquida desde una temperatura inicial, atomizar la sustancia líquida, de modo que se obtenga un tamaño de partícula predeterminado, congelar la sustancia líquida atomizada (ALS) a por debajo de su temperatura eutéctica, conducir la ALS congelada a una cámara de secado, que se mantiene bajo vacío, conducir la ALS a través de la cámara de secado, y calentar la ALS congelada mediante una gradiente de temperatura, mientras se desplaza a través de la cámara de secado, de modo que la graduación de temperatura pueda efectuar la sublimación y secado, evitando o minimizando al mismo tiempo que ocurra cualquier deterioro por calor a la ALS, a medida que pasa a través de la cámara a un punto de recolección de la ALS seca.

El paso de enfriar una sustancia líquida puede tener lugar en un enfriador para reducir la sustancia líquida desde una temperatura inicial a una temperatura más baja. Por ejemplo, esto puede significar que la sustancia líquida se enfría desde condiciones ambientales y/o la temperatura del equipo de acondicionamiento de almacenamiento a una temperatura más baja. Este paso de enfriamiento es opcional; no obstante, puede ayudar y reducir la carga de enfriamiento requerida por el congelador por atomización.

El enfriamiento puede obtenerse por cualquier método o aparato apropiado, que puedan reducir la temperatura de una sustancia desde una temperatura inicial a una temperatura más baja. Preferiblemente el enfriamiento puede lograrse por cualquier unidad adecuada de transferencia de calor; éstas pueden ser por ejemplo refrigeradores, intercambiadores de calor de placas, intercambiadores de calor de envolvente y tubos, bombas de calor, aparatos de convección de aire frío, torres de enfriamiento gas-líquido y otros aparatos similares de enfriamiento apropiados. El congelador por atomización es un aspecto especialmente importante de la configuración de procesamiento continuo, en la cual se desea una rápida reducción de la temperatura a por debajo de la temperatura eutéctica de la sustancia, así como atomización de la sustancia líquida. Preferiblemente, la atomización de la sustancia líquida puede inducirse por una variedad de dispositivos de alimentación tales como, boquilla de líquido único (tipo presión), boquilla de dos líquidos (tipo neumático), boquillas centrífugas (disco giratorio), ultrasónicas, y pueden emplearse varios otros atomizadores rotatorios y técnicas de atomización de aire. El enfriamiento de la ALS puede conseguirse mediante el uso de un enfriador capaz de congelar la ALS. Preferiblemente la atomización y enfriamiento de la sustancia líquida tienen ambas lugar en un congelador por atomización.

Hay un número de ventajas del congelamiento por atomización, especialmente porque los particulados formados pueden producirse con tamaños de partículas específicos o predeterminados, y la especificación, o la calidad del particulado, permanece sustancialmente constante de principio a fin de toda la operación del congelador por atomización. La operación del congelador por atomización es idealmente continua y adaptable a control automático total, donde los tiempos de respuesta son rápidos. El congelamiento por atomización es una aplicación útil a sustancias sensibles al calor y resistentes al calor. No obstante, se estima que puede requerirse igualmente un procesamiento semi-continuo.

Ocurre un cambio de fase en la ALS cuando se reduce la temperatura de la ALS, es decir, en la cual los líquidos se congelan o reducen a por debajo de su temperatura eutéctica (es decir, sólidos). Líquidos pueden sublimarse de la ALS congelada en un estado posterior en el proceso, luego de exponerse a una gradiente de temperatura y fuente de energía.

Los ciclones son uno de los principales métodos usados para separar fases gas-sólido, y pueden proporcionar una separación eficiente. ALS congelada y gases se separan, reciclándose el gas para enfriar la sustancia líquida, y conduciéndose la ALS congelada (sólida) a través de la cámara de secado. La sublimación es el cambio de fase de líquidos congelados (sólido) a gas (vapor líquido), por medio de evaporación de líquido de la ALS. Idealmente, la sublimación y/o secado de la ALS congelada tiene lugar en la superficie. Más preferiblemente la superficie puede ser una superficie vibradora. Aún más preferiblemente, la superficie vibradora es una bandeja vibradora, en la cual las vibraciones se producen por medios neumáticos y/o mecánicos y/o eléctricos. Es ventajoso inducir y/o proveer cierto movimiento de vibración de la ALS congelada a secarse, para intensificar y promover la transferencia de calor a las partículas atomizadas.

La sublimación (es decir, el secado de la ALS y la eliminación de líquido "libre") puede iniciarse y efectuarse por la energía emitida de una fuente de energía, tal como un dispositivo infrarrojo, con lo cual la bandeja de secado vibradora mejora preferiblemente las características de transferencia de calor a la ALS y permite una fase de secado más uniforme y controlada. La fuente de energía capaz de calentar la ALS puede ser cualquier fuente de energía apropiada para inducir sublimación de los líquidos congelados de la ALS. Tales fuentes de energía adecuadas pueden incluir por ejemplo: dispositivos emisores infrarrojos, microondas, calentadores radiantes, calentadores de convección, y cualquier dispositivo que provea energía suficiente a la ALS para inducir sublimación de los líquidos "libres" congelados de la ALS.

La eliminación de líquidos de sustancias para formar un producto seco, controlando las velocidades de flujo del aire, temperatura y presión reduce el contenido de humedad de sustancias y contribuye a inhibir el crecimiento microbiano, que puede causar descomposición y deterioro. La eliminación de humedad reduce también el peso, lo cual es de interés y consideración significativos para el embarque y transporte.

El pretratamiento químico de algunas sustancias puede contribuir aún más a aumentar la vida útil del producto. Estos pretratamientos pueden incluir sustancias para mejorar la conservación.

A menudo la deshidratación a una escala industrial requiere que un producto de suministro se reduzca en tamaño y/o se particula para permitir características mejoradas de procesamiento. El secado por congelamiento es el secado de material en un estado congelado y, preferiblemente, la fase de secado tiene lugar a una presión absoluta, que fácilmente puede permitir que el hielo experimente un cambio de fase directamente de sólido a vapor. Preferiblemente, en la presente invención el producto secado por congelamiento sale sustancialmente en buen estado de la fase de cámara de secado.

En un proceso mejorado de secado de los aspectos primero y segundo, la sustancia líquida puede atomizarse en un congelador por atomización para conseguir un tamaño de partícula de sustancialmente 500 micrones o más. Alternativamente, el tamaño de partícula obtenido puede ser menos de sustancialmente 500 micrones, o incluso menos de de sustancialmente 200 micrones. La atomización de la sustancia líquida es deseable, de modo que la sustancia líquida atomizada pueda reducirse rápidamente en temperatura a por debajo de su temperatura eutéctica. Además, la atomización de la sustancia líquida y el congelamiento subsiguiente permite procesar un producto particulado, lo cual posibilita características mejoradas de control de secado y transferencia de calor.

El aparato o congelador por atomización puede ser de una configuración contracorriente, aunque puede ser también de configuración de la misma corriente, en la cual el congelador por atomización puede utilizar un gas frío para efectuar un cambio de fase de los líquidos en la ALS. El gas frío puede efectuar un cambio de fase [que] ocasiona el congelamiento de líquidos en la sustancia líquida atomizada (es decir, la ALS se reduce a una temperatura por debajo de su temperatura eutéctica). El aire frío utilizado por el congelador por atomización para efectuar el cambio de fase puede ser aire frío, aunque puede usarse cualquier gas adecuado para efectuar un cambio de fase de líquidos contenidos en la ALS.

El gas frío empleado puede estar a sustancialmente 0°C o menos, incluso más preferiblemente el gas frío puede estar a menos de sustancialmente -20°C.

La ALS una vez congelada/reducida a por debajo de su temperatura eutéctica puede conducirse entonces a un separador, y la conducción puede tener lugar neumáticamente. El separador puede ser un dispositivo de separación gas-sólido, por ejemplo, un ciclón.

El gas separado del ciclón puede realimentarse y/o refrigerarse para uso en el congelador por atomización. Puede reciclarse asimismo al congelador por atomización para reducir la carga de enfriamiento en el evaporador. El evaporador se usa para enfriar el gas, el cual se emplea para inducir un cambio de fase de líquidos en la ALS. Sin embargo, puede utilizarse cualquier aparato apropiado de enfriamiento de gas, usándose un evaporador como ejemplo en este caso. Los aparatos de enfriamiento adecuados pueden incluir refrigeradores, intercambiadores de calor de placas, intercambiadores de calor de envolvente y tubos, y otras unidades de transferencia de calor capaces de enfriar un gas a una temperatura, que pueda suministrarse a un congelador por atomización para forzar un cambio de fase de líquidos en la ALS al estado sólido (congelado).

Preferiblemente uno o más cierres de vacío y/o aire se encuentran situados entre la salida del ciclón de la ALS congelada y la entrada de la cámara de secado, o algún tipo de dispositivo de sellamiento de presión. Es ventajoso proveer uno o más dispositivos de cierre de presión para mantenimiento del vacío en la cámara de secado. Sin el uso de estos dispositivos de cierre de presión no puede obtenerse un vacío apropiado en la cámara de secado. Cierres de presión, tales como válvulas, pueden ser particularmente apropiados, aunque puede utilizarse cualquier dispositivo adecuado para sellar la cámara de secado de vacío y garantizar que la cámara de secado mantenga su vacío.

En incorporaciones adicionales puede haber un sistema de refrigeración, que se emplea para mantener la ALS congelada a una temperatura por debajo de su temperatura eutéctica durante el transporte desde el ciclón a la cámara de secado de vacío. Es ventajoso que la ALS congelada permanezca a una temperatura por debajo de su temperatura eutéctica para minimizar cualquier degradación o deterioro del producto que pueda ocurrir, si la ALS sube por encima de su temperatura eutéctica.

El vacío de la cámara de secado de vacío se crea por un dispositivo apropiado de reducción de presión.

El vacío creado por tales dispositivos adecuados de reducción de presión puede ser sustancialmente 600 micrómetros Hg a presión seleccionada o menos. Más preferiblemente el vacío creado puede estar en el rango de sustancialmente 200-400 micrómetros Hg de presión absoluta.

El transporte o conducción de la ALS congelada a través de la cámara de vacío puede tener lugar en una superficie vibradora, donde la superficie vibradora es una bandeja vibradora. La bandeja vibradora puede accionarse por medios neumáticos, eléctricos y/o mecánicos para impartir la vibración a la bandeja.

La bandeja vibradora conduce la ALS congelada a través de una gradiente de temperatura para efectuar sublimación, retirándose el vapor producido por sublimación de la cámara de secado por uno o más condensadores o similares.

La gradiente de temperatura a través de la bandeja de secado se provee por una fuente de energía, tal como un dispositivo emisor infrarrojo. No obstante, se preve el uso de otras fuentes de energía, por ejemplo calentadores óhmicos y dispositivos microondas.

El producto generado por un proceso y aparato mejorados de secado está sustancialmente libre de líquido o, en otra forma, sustancialmente seco o, por lo menos, sustancialmente reducido en contenido de líquido. Se desea un aparato para secado mejorado, que facilita uno o más de los siguientes pasos de enfriar una sustancia líquida desde una temperatura inicial a una temperatura más baja, atomizar una sustancia líquida, de modo que pueda obtenerse un tamaño de partícula predeterminado, enfriar la sustancia líquida atomizada (ALS) a por debajo de su temperatura eutéctica, conducir la ALS congelada a una cámara de secado, que puede mantenerse bajo vacío, y calentar la ALS congelada con el fin de efectuar sublimación y secado por congelamiento y, seguidamente, recoger la ALS seca de la cámara de secado. En una incorporación alternativa el aparato se define como un aparato para un proceso de secado mejorado, el cual puede facilitar uno o más de los pasos de enfriar la sustancia líquida desde una temperatura inicial, atomizar la sustancia líquida con el fin de poder obtener un tamaño de partícula predeterminado, enfriar la sustancia líquida atomizada (ALS) a por debajo de su temperatura eutéctica, conducir la ALS congelada a una cámara de secado, que mantiene bajo vacío, conducir la ALS a través de la cámara de secado y calentar la ALS congelada mediante una gradiente de temperatura, mientras se desplaza a través de la cámara de secado, de manera que la gradiente de temperatura pueda efectuar sublimación y secar, previniendo o minimizando al mismo tiempo cualquier deterioro por calor que ocurra a la ALS, a medida que pasa a través de la cámara a un punto de recolección de la ALS seca. Sería preferible, pero no necesariamente esencial, que los pasos del proceso y metodología puedan conectarse para formar un proceso continuo. Un proceso continuo puede tener un número de ventajas.

Adicionalmente, un proceso que se configura para operarse sobre una base continua puede ser preferible, ya que un sistema continuo tiende a optimizarse más fácilmente en cuanto a productividad, rendimiento más alto, tiempo de procesamiento reducido por unidad producida, calidad mejorada como resultado de controles optimizados, un proceso generalmente más eficiente, reducción del manejo humano y/o contacto con sustancias, que pueden contaminar equipo y/o sustancias; y la reducción de la mano de obra manual humana preferiblemente reduce también la probabilidad de lesión. Además, el procesamiento continuo puede permitir equipo diseñado para una solución optimizada de procesamiento continuo y producción de productos secados por congelamiento.

La cámara de secado de vacío se diseña para operar a presiones absolutas en el rango de 200-400 micrómetros de Hg. El estado de vacío de la cámara de secado permite el cambio de fase preferencial (sublimación) de los líquidos congelados a la fase gas/vapor. El material de sublimación (vapor) puede eliminarse preferiblemente por el uso de un condensador. En una incorporación preferida la cámara de secado se opera continuamente, y puede conectarse con congeladores por atomización adicionales, dispositivos de separación (ciclones), sistemas de empaque a operarse sobre una base por lotes, semi-continua o continua.

En una incorporación preferida de la presente invención, la ALS puede caer sobre una bandeja vibradora neumática inclinada, (aunque pueden emplearse otros sistemas de bandeja vibradora) para conducir la ALS a través de una gradiente de temperatura para efectuar sublimación y descarga de producto seco al final del secador. El producto seco sale a través del cierre de vacío de descarga (que garantiza que el vacío se mantenga) hacia fases adicionales de procesamiento. Por ejemplo, fases adicionales de procesamiento pueden incluir sistemas de empaque discreto.

Es preferible que la temperatura de la superficie de la bandeja se controle en toda su longitud para garantizar un secado completo, sin deterioro de los productos.

El secado puede definirse como la aplicación de energía bajo condiciones controladas, para eliminar por evaporación la mayor parte del agua normalmente presente en una sustancia. El propósito principal de la deshidratación es ampliar la vida útil de sustancias mediante una reducción del contenido de agua. El secado puede causar detrimento en la calidad alimenticia y el valor nutritivo del alimento. El agua juega un papel importante en la estabilidad de alimentos frescos, congelados y secados, ya que actúa como un disolvente para una reacción química, microbiológica y enzimática.

Un secador puede incluir una bandeja vibradora, un medio de reducción de presión, un puerto de entrada de material, un puerto de salida de material y una fuente de calor. La bandeja vibradora provee una superficie vibradora (sobre la cual ocurre la sublimación), que conduce ALS congelada del puerto de entrada de material al puerto de salida de material. El puerto de entrada de material es el punto de ingreso de la ALS congelada al secador, y el puerto de salida de material es el punto de egreso de la ALS seca (pos-sublimación).

Se utiliza una fuente de calor para inducir la sublimación de líquidos congelados de la ALS congelada, a lo largo de la bandeja vibradora.

Por lo tanto, es ventajoso proveer una serie de fases de tratamiento en las cuales una sustancia líquida pueda procesarse y secarse sustancialmente o tener un contenido líquido reducido. Los pasos descritos de enfriar opcionalmente una sustancia líquida, atomizar la sustancia líquida, enfriar la ALS para iniciar un cambio de fase de los líquidos en la ALS, separar opcionalmente del gas de enfriamiento la ALS cambiada de fase, conducir la ALS cambiada de fase a una cámara de secado, y eliminación de líquidos de la ALS por sublimación pueden designarse como una serie de fases de tratamiento para proveer una sustancia con contenido de líquido/humedad sustancialmente reducido.

Sin embargo, se considera asimismo que puede prescindirse de los pasos de pretratamiento y la sustancia líquida puede insertarse directamente en la cámara de vacío (la cual se mantiene a una presión de vacío, que puede estar por debajo de la presión de punto triple de la sustancia líquida que se está procesando). Esta inyección directa puede resultar en la evaporación instantánea de una porción del líquido, reduciéndose por consiguiente en temperatura el resto de la sustancia líquida, de modo que resulten partículas de sustancia líquida congelada. Estas partículas congeladas pueden tratarse entonces en la cámara de secado para inducir sublimación del líquido restante, con el fin de obtener una sustancia líquida sustancialmente más seca.

Pueden considerarse las fases de pretratamiento para mejorar el proceso global, aunque pueden suprimirse donde la inyección directa de la sustancia líquida a la cámara de secado de presión de vacío es una opción.

Los expertos en el arte observarán, entenderán y podrán calcular las dimensiones de las bombas, calentadores, motores, boquillas de aspersión o atomización requeridas para realizar la presente invención, y se observa que existen numerosas variables que dictarán o influirán en las dimensiones de estas clases de componentes. Debe observarse asimismo que el diseño de una cámara de vacío estará al alcance de una persona experta en el arte – no obstante, es la combinación y las ventajas dadas a conocer más arriba

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las que hacen apropiada esta invención. Los aspectos de la presente invención se han descrito a modo de ejemplo únicamente, y debe observarse que pueden introducirse modificaciones y adiciones a ellos, sin apartarse del ámbito de la misma, como se define en las reivindicaciones adjuntas.

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LAS REIVINDICACIONES QUE DEFINEN LA INVENCIÓN SON:

1. Un método de secar o concentrar una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella, el cual incluye los pasos de:
   mantener una cámara a una temperatura y presión por debajo del punto triple de la sustancia líquida,
   inyectar y/o atomizar la sustancia líquida a la cámara para generar una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),
   recoger la porción FLS como una capa en una superficie, y
   conducir la porción FLS recogida en la superficie,
   en donde la superficie conduce la porción FLS recogida a una velocidad que controla el espesor de la capa de FLS recogida en la superficie.

2. Un método como se reivindica en la reivindicación 1, en donde la velocidad de conducción logra sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

3. Un método como se reivindica en la reivindicación 1 o la reivindicación 2, en donde la FEL se condensa por un dispositivo condensador.

4. Un método como se reivindica en una cualquiera de las reivindicaciones 1 a 3, en donde la FEL se extrae de dicha cámara.

5. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde inyectar y/o atomizar la sustancia líquida incluye atomización para obtener un tamaño de partícula predeterminado de FLS.

6. Un método como se reivindica en una cualquiera de las

reivindicaciones precedentes, en donde la inyección y/o atomización de la sustancia líquida se realiza por una o más boquillas.

7. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la velocidad de conducción de FLS sobre dicha superficie obtiene sustancialmente un espesor de monocapa de la FLS sobre dicha superficie.

8. Un método como se reivindica en la reivindicación 7, en donde la monocapa puede tener el espesor de una capa única del tamaño de partícula predeterminado de la porción FLS.

9. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es sustancialmente 500 μm o más.

10. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es menos de sustancialmente 500 μm.

11. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es menos de sustancialmente 200 μm.

12. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la superficie conduce la porción FLS alejándola de las una o más boquillas.

13. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde el método incluye el paso de exponer la capa recogida de porción FLS a un medio de calefacción, induciendo sustancialmente en esa forma sublimación y generación de una segunda porción de sustancia líquida evaporada ("porción SEL") y

un producto.

14. Un método como se reivindica en la reivindicación 13, en donde la porción SEL se condensa por un dispositivo condensador.

15. Un método como se reivindica en la reivindicación 13 o la reivindicación 14, en donde la porción SEL se extrae de la cámara.

16. Un método como se reivindica en una cualquiera de las reivindicaciones 13 a 15, en donde el producto se extrae de la superficie de conducción por un dispositivo de extracción del producto.

17. Un método como se reivindica en una cualquiera de las reivindicaciones 13 a 16, en donde el producto se extrae de la cámara a través del puerto de salida.

18. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde el método se utiliza para procesar una sustancia fluidizada, o una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella.

19. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, donde la sustancia se selecciona de una o más de las siguientes: una suspensión de café, leche líquida, jugos de frutas y/o vegetales.

20. Un aparato para secar o concentrar una sustancia líquida, el cual comprende:
una cámara y un dispositivo de reducción de presión de la cámara, y
uno o más puertos de inyección a través de los cuales se inyecta la sustancia líquida a la cámara,

una superficie de recolección que recoge una porción de sustancia líquida congelada de la sustancia líquida,

en donde el dispositivo de reducción de presión mantiene la cámara a una presión por debajo de por lo menos la presión de punto triple de la sustancia líquida, para hacer que la sustancia líquida inyectada se separe en una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),

de modo que, en uso, la porción FLS se acumule como una capa en la superficie de recolección y se conduzca lejos de los uno o más puertos de inyección a una velocidad que permita controlar el espesor de la capa de la porción FLS.

21. Un aparato como se reivindica en la reivindicación 20, en donde la velocidad de conducción obtiene sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

22. Un aparato como se reivindica en la reivindicación 20 o la reivindicación 21, en donde la porción FEL se condensa por un dispositivo condensador.

23. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 22, en donde dicha FEL se extrae de dicha cámara.

24. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a la reivindicación 23, en donde el uno o más puertos de inyección comprenden por lo menos una boquilla de aspersión o atomización.

25. Un aparato como se reivindica en la reivindicación 24, en donde la boquilla o boquillas determinan sustancialmente el tamaño de la

porción FLS generada para lograr un tamaño de partícula predeterminado.

26. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 25, en donde el dispositivo de reducción de presión es una bomba de evacuación de gas.

27. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 26, en donde el dispositivo condensador es un serpentín o serpentines refrigerados.

28. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 27, en donde el dispositivo condensador se enfría con un refrigerante.

29. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 28, en donde la superficie de recolección incluye uno, o una combinación, de los siguientes transportadores mecánicos: una configuración de cinta sinfín, una bandeja inclinada para favorecer que la porción de sustancia líquida congelada se deslice desde el uno o más puertos de inyección, una bandeja vibradora.

30. Un aparato como se reivindica en la reivindicación 29, en donde la bandeja inclinada y/o bandeja vibradora incluye una superficie de fricción reducida.

31. Un aparato como se reivindica en la reivindicación 30, en donde la superficie de fricción reducida comprende politetrafluroetileno (PTFE).

32. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 31, en donde la capa de la porción FLS sobre

dicha superficie de recolección se expone a un medio de calefacción, a medida que se conduce lejos del uno o más puertos de inyección.

33. Un aparato como se reivindica en la reivindicación 32, en donde el medio de calefacción sustancialmente induce sublimación de la capa de porción FLS sobre dicha superficie para formar una segunda porción de sustancia líquida evaporada ("SEL") y un producto.

34. Un aparato como se reivindica en la reivindicación 32 o la reivindicación 33, en donde el medio de calefacción es una, o una combinación, de las siguientes fuentes de energía: lámparas infrarrojas, lámparas halógenas, lámparas incandescentes, microondas, o calentamiento óhmico de dicha superficie.

35. Un aparato como se reivindica en la reivindicación 33 o la reivindicación 34, en donde un dispositivo de extracción del producto se emplea para extraer el producto de la superficie de recolección.

36. Un aparato como se reivindica en la reivindicación 35, en donde el dispositivo de extracción del producto es un medio raspador y/o un medio de arrastre de cepillo.

37. Un aparato como se reivindica en la reivindicación 36, en donde el medio raspador contacta sustancialmente la superficie, con el producto sobre ella, y desplaza el producto de la superficie al puerto de salida.

38. Un aparato como se reivindica en la reivindicación 36, en donde el medio de arrastre de cepillo es un cepillo giratorio o cepillo fijo, que contacta sustancialmente la superficie, con el producto sobre ella, y dirige el producto de la superficie al puerto de salida.

39. Un aparato como se reivindica en una cualquiera de las

reivindicaciones 33 a 37, en donde el producto se extrae a través de un puerto de salida de la cámara.

40. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 39, en donde la cámara se mantiene a una presión de sustancialmente 611.3 Pa o menos.

41. Un proceso de secado para una sustancia líquida, el cual comprende los pasos de:
atomizar la sustancia líquida,
enfriar la sustancia líquida atomizada (ALS) para iniciar un cambio de fase,
conducir la ALS a una cámara de secado bajo vacío,
calentar la ALS con el fin de efectuar sustancialmente sublimación y, luego, recoger la ALS sustancialmente seca de la cámara.

42. Un proceso de secado como se reivindica en la reivindicación 41, en donde la sustancia líquida se enfría desde una temperatura inicial antes de atomizarla.

43. Un proceso de secado como se reivindica en la reivindicación 41 o la reivindicación 42, en donde la ALS conducida a la cámara de secado se conduce también a través de la cámara de secado.

44. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 43, en donde la ALS se calienta mediante una gradiente de temperatura, mientras se desplaza a través de la cámara de secado, de modo que la gradiente de temperatura efectúa sustancialmente sublimación.

45. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 42 a 44, en donde el paso de enfriar una sustancia

líquida tiene lugar en un enfriador, para reducir la sustancia líquida desde una temperatura inicial a una temperatura más baja.

46. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 45, en donde atomizar la sustancia líquida alcanza un tamaño de partícula predeterminado.

47. Un proceso de secado como se reivindica en la reivindicación 46, en donde el tamaño de partícula predeterminado es sustancialmente 500 µm o más.

48. Un proceso de secado como se reivindica en la reivindicación 46, en donde el tamaño de partícula predeterminado es menos de sustancialmente 500 µm.

49. Un proceso de secado como se reivindica en la reivindicación 46, en donde el tamaño de partícula predeterminado es menos de sustancialmente 200 µm.

50. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 49, en donde un congelador por atomización utiliza un gas frío para efectuar un cambio de fase de los líquidos en la sustancia líquida atomizada (ALS).

51. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 42 a 50, en donde el enfriamiento se realiza con contacto directo o indirecto con un fluido frío.

52. Un proceso de secado como se reivindica en la reivindicación 51, en donde dicho fluido frío es aire.

53. Un proceso de secado como se reivindica en la reivindicación 51 o la

reivindicación 52, en donde dicho fluido frío está sustancialmente a 0°C o menos.

54. Un proceso de secado como se reivindica en la reivindicación 51 o la reivindicación 52, en donde dicho fluido frío está a menos de sustancialmente -20°C.

55. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 50 a 54, en donde dicho congelador por atomización opera en una configuración contracorriente.

56. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 50 a 54, en donde dicho congelador por atomización opera en una configuración de la misma corriente.

57. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 56, en donde la ALS se reduce a una temperatura por debajo de su temperatura eutéctica.

58. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 57, en donde la ALS se conduce a un separador.

59. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 57, en donde dicha ALS se conduce neumáticamente a un separador.

60. Un proceso de secado como se reivindica en la reivindicación 58 o la reivindicación 59, en donde dicho separador es un dispositivo de separación gas-sólido.

61. Un proceso de secado como se reivindica en la reivindicación 60, en donde dicho dispositivo de separación gas-sólido es un ciclón.

al

62. Un proceso de secado como se reivindica en la reivindicación 60 o la reivindicación 61, en donde el gas separado en el dispositivo de separación gas-sólido se realimenta y/o refrigera para uso en el paso de enfriamiento.

63. Un proceso de secado como se reivindica en la reivindicación 61 o la reivindicación 62, en donde uno o más cierres de vacío y/o aire están presentes entre una salida del ciclón AFS y una entrada de la cámara de secado.

64. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 60 a 63, en donde sólidos separados de dicho dispositivo de separación gas-sólido ingresan a una cámara de secado de vacío.

65. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 64, en donde el vacío de dicha cámara de secado de vacío se crea por un dispositivo de reducción de presión.

66. Un proceso de secado como se reivindica en la reivindicación 65, en donde el vacío creado por el dispositivo de reducción de presión es sustancialmente 600 micrómetros Hg de presión absoluta o menos.

67. Un proceso de secado como se reivindica en la reivindicación 65, en donde el vacío creado por el dispositivo de reducción de presión está en el rango de sustancialmente 200-400 micrómetros Hg de presión absoluta.

68. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 67, en donde dicha ALS se conduce mediante una superficie.

69. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 68, en donde dicha ALS se conduce mediante una superficie vibradora.

70. Un proceso de secado como se reivindica en la reivindicación 69, en donde dicha superficie vibradora es una bandeja vibradora.

71. Un proceso de secado como se reivindica en la reivindicación 69 o la reivindicación 70, en donde dicha bandeja vibradora se acciona neumática y/o mecánica y/o eléctrica y/o magnéticamente.

72. Un proceso de secado como se reivindica en la reivindicación 70 o la reivindicación 71, en donde dicha bandeja vibradora conduce la ALS a través de una gradiente de temperatura para efectuar sustancialmente sublimación.

73. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 72, en donde un vapor producido por sublimación se extrae de la cámara de secado.

74. Un proceso de secado como se reivindica en la reivindicación 73, en donde vapor producido por sublimación se extrae de la cámara de secado por uno o más condensadores.

75 Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 74, en donde la ALS se calienta mediante una gradiente de temperatura, mientras se desplaza a través de la cámara de secado, de modo que la gradiente de temperatura efectúa sustancialmente sublimación.

76. Un proceso de secado como se reivindica en una cualquiera de las

reivindicaciones 41 a 75, en donde dicha gradiente de temperatura se provee por una fuente de energía.

77. Un proceso de secado como se reivindica en la reivindicación 76, en donde dicha fuente de energía es un dispositivo emisor infrarrojo y/o un dispositivo emisor microondas y/o un calentador óhmico.

78. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 77, en donde atomizar la sustancia líquida alcanza un tamaño de partícula predeterminado.

79. Un proceso de secado como se reivindica en la reivindicación 78, en donde el tamaño de partícula predeterminado es sustancialmente 500 µm o más.

80. Un proceso de secado como se reivindica en la reivindicación 78, en donde el tamaño de partícula predeterminado es menos de sustancialmente 500 µm.

81. Un proceso de secado como se reivindica en la reivindicación 78, en donde el tamaño de partícula predeterminado es menos de sustancialmente 200 µm.

82. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 60 a 81, en donde se emplea un sistema de refrigeración para mantener ALS a una temperatura por debajo de su temperatura eutéctica durante el transporte desde el dispositivo de separación gas-sólido a la cámara de secado de vacío.

83. Un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 41 a 82, en donde un producto generado sustancialmente reducido está libre de líquido, comparado con la

sustancia líquida.

84. Un aparato para un proceso de secado de una sustancia líquida, el cual comprende:
un atomizador capaz de atomizar una sustancia líquida,
un enfriador capaz de enfriar la sustancia líquida atomizada (ALS) para iniciar un cambio de fase,
un transportador mecánico capaz de conducir la ALS a una cámara de secado mantenida bajo vacío,
una fuente de energía capaz de calentar la ALS, con el fin de efectuar sublimación y secado por congelamiento, y
un recolector capaz de recolectar la ALS seca.

85. Un aparato para un proceso de secado como se reivindica en la reivindicación 84, en donde antes del atomizador de la sustancia líquida se provee un enfriador, capaz de enfriar una sustancia líquida desde una temperatura inicial a una temperatura más baja.

86. Un aparato para un proceso de secado como se reivindica en la reivindicación 84 o la reivindicación 85, en donde el aparato incluye un transportador mecánico capaz de conducir la ALS a través de la cámara de secado.

87. Un aparato para un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 84 a 86, en donde el aparato incluye una fuente de energía capaz de calentar la ALS mediante una gradiente de temperatura, mientras se desplaza a través de la cámara de secado, de modo que la gradiente de temperatura pueda efectuar sublimación.

88. Un aparato para un proceso de secado como se reivindica en la reivindicación 87, en donde la gradiente de temperatura

sustancialmente impide o sustancialmente minimiza el deterioro por calor que ocurre a la ALS, a medida que pasa a través de la cámara.

89. Un aparato para un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 84 a 88, en donde atomizar la sustancia líquida obtiene un tamaño de partícula predeterminado.

90. Un aparato para un proceso de secado como se reivindica en la reivindicación 89, en donde el tamaño de partícula predeterminado es sustancialmente 500 µm o más.

91. Un aparato para un proceso de secado como se reivindica en la reivindicación 89, en donde el tamaño de partícula predeterminado es menos de sustancialmente 500 µm.

92. Un aparato para un proceso de secado como se reivindica en la reivindicación 89, en donde el tamaño predeterminado es menos de sustancialmente 200 µm.

93. Un aparato para un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 84 a 92, en donde un congelador por atomización utiliza un gas frío para efectuar un cambio de fase de los líquidos en la sustancia líquida atomizada (ALS).

94. Un aparato para un proceso de secado como se reivindica en la reivindicación 93, en donde dicho gas frío es aire.

95. Un aparato para un proceso de secado como se reivindica en la reivindicación 93 o la reivindicación 94, en donde dicho gas frío está sustancialmente a 0°C o menos.

96. Un aparato para un proceso de secado como se reivindica en la

reivindicación 93 o la reivindicación 94, en donde dicho gas frío está a menos de sustancialmente -20°C.

97. Un aparato para un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 84 a 96, en donde el vacío de dicha cámara de secado de vacío se crea por un dispositivo de reducción de presión.

98. Un aparato para un proceso de secado como se reivindica en la reivindicación 97, en donde el vacío creado por el dispositivo de reducción de presión es sustancialmente 600 micrómetros Hg de presión absoluta o menos.

99. Un aparato para un proceso de secado como se reivindica en la reivindicación 97, en donde el vacío creado por el dispositivo de reducción de presión está en el rango de sustancialmente 200-400 micrómetros Hg de presión absoluta.

100. Un aparato para un proceso de secado como se reivindica en una cualquiera de las reivindicaciones 84 a 99, en donde dicha ALS se conduce mediante una superficie vibradora.

101. Un aparato para un proceso de secado como se reivindica en la reivindicación 100, en donde dicha superficie vibradora es una bandeja vibradora.

102. Un aparato para un proceso de secado como se reivindica en la reivindicación 101, en donde dicha bandeja vibradora se acciona neumática y/o mecánica y/o eléctrica y/o magnéticamente.

103. Un aparato para un proceso de secado como se reivindica en la reivindicación 84 o la reivindicación 102, en donde la ALS se conduce

a través de una gradiente de temperatura para efectuar sustancialmente sublimación.

104. Un aparato para un proceso de secado como se reivindica en la reivindicación 84 o la reivindicación 103, en donde el vapor producido por sublimación se extrae de la cámara de secado por uno o más condensadores.

105. Un aparato para un proceso de secado como se reivindica en la reivindicación 103, en donde dicha gradiente de temperatura se provee por una fuente de energía.

106. Un aparato para un proceso de secado como se reivindica en la reivindicación 105, en donde dicha fuente de energía es un dispositivo emisor infrarrojo y/o un dispositivo emisor microondas y/o un calentador óhmico.

107. Una cámara de secado que comprende:
un dispositivo capaz de hacer vibrar una bandeja,
un dispositivo apropiado para reducir la presión en dicha cámara,
un puerto de salida de material de dicha cámara, y
una fuente de calor adaptada para actuar sobre dicha bandeja.

98

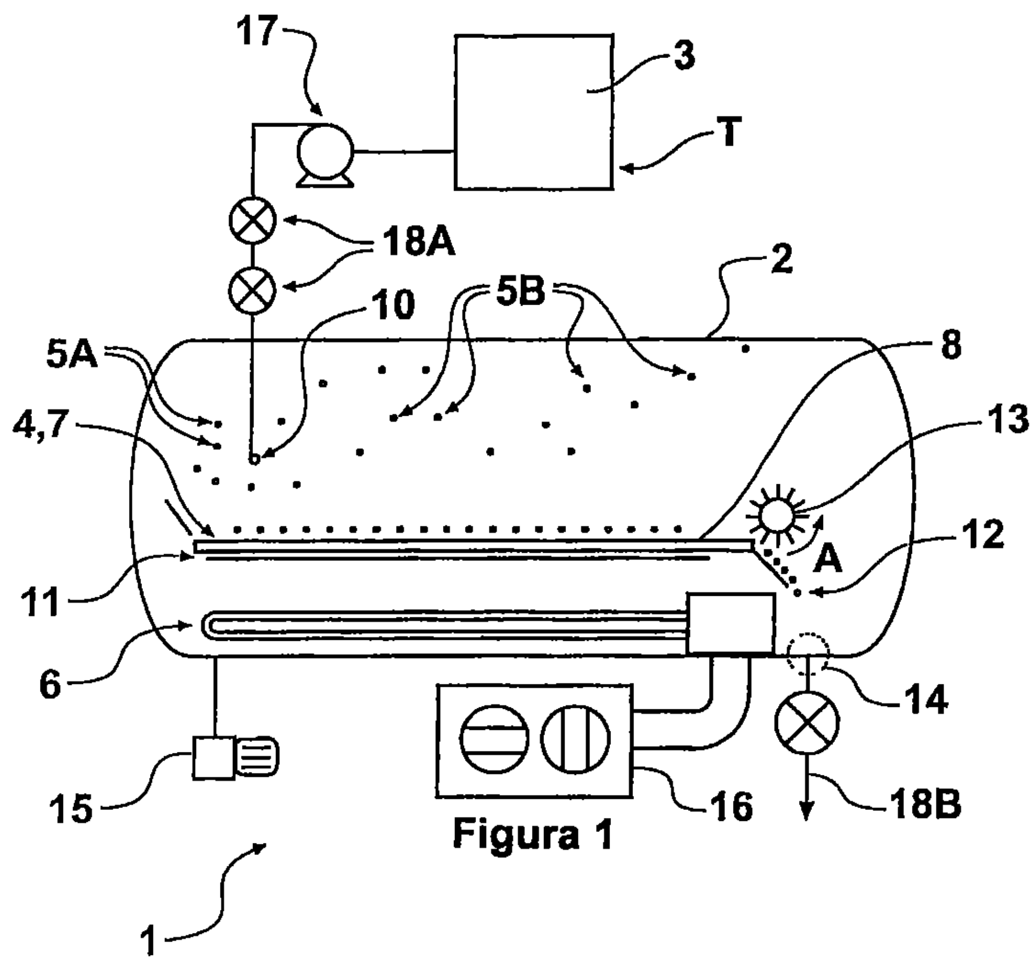

Figura 1

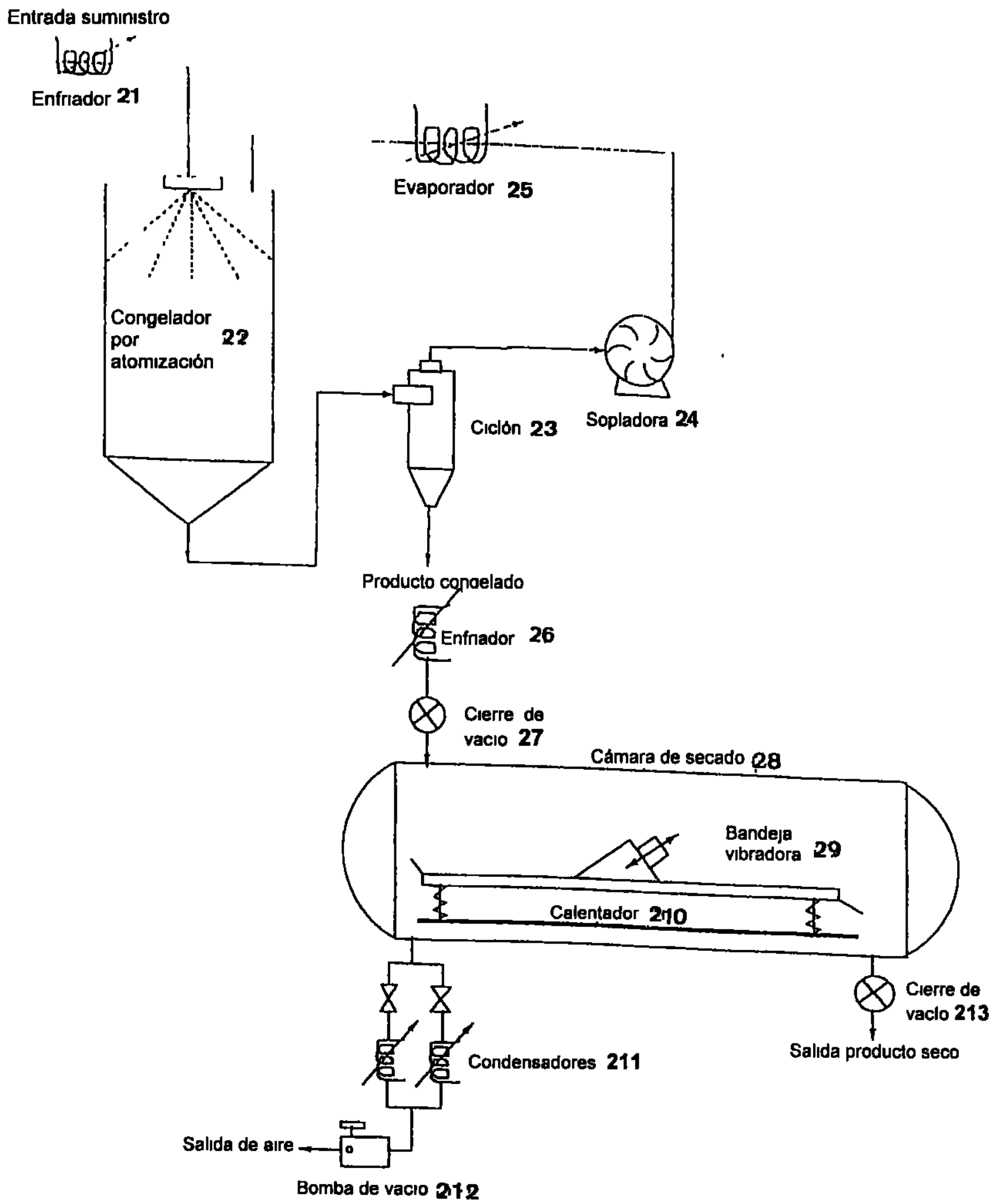
Entrada suministro
Enfriador 21
Evaporador 25
Congelador por atomización 22
Sopladora 24
Ciclón 23
Producto congelado
Enfriador 26
Cierre de vacio 27
Cámara de secado 28
Bandeja vibradora 29
Calentador 210
Cierre de vacio 213
Salida producto seco
Condensadores 211
Salida de aire
Bomba de vacio 212

Figura 2

100

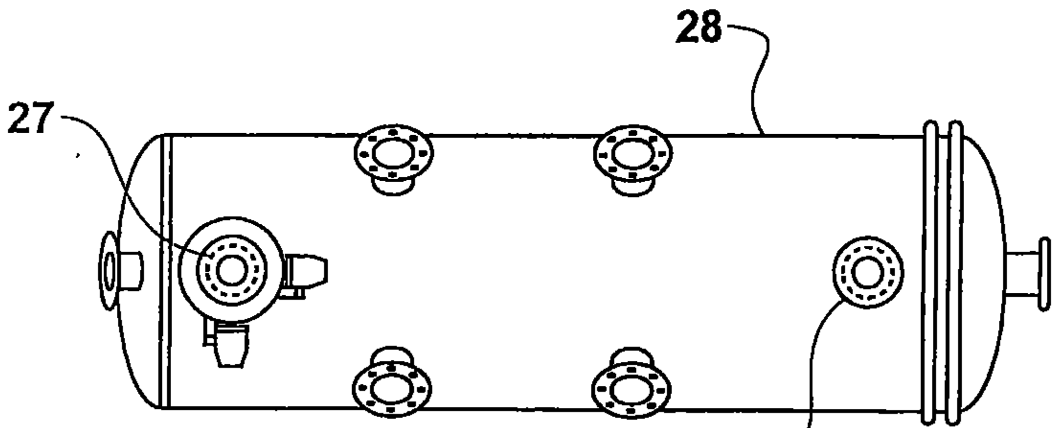

Figura 3

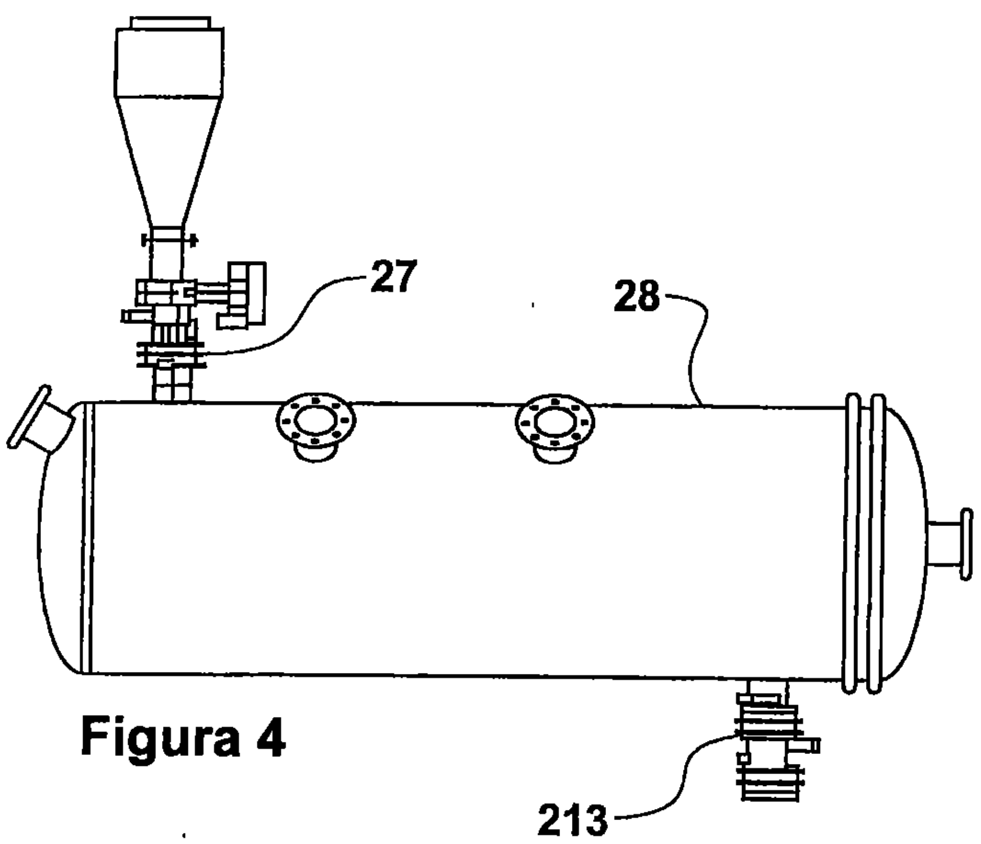

Figura 4

101

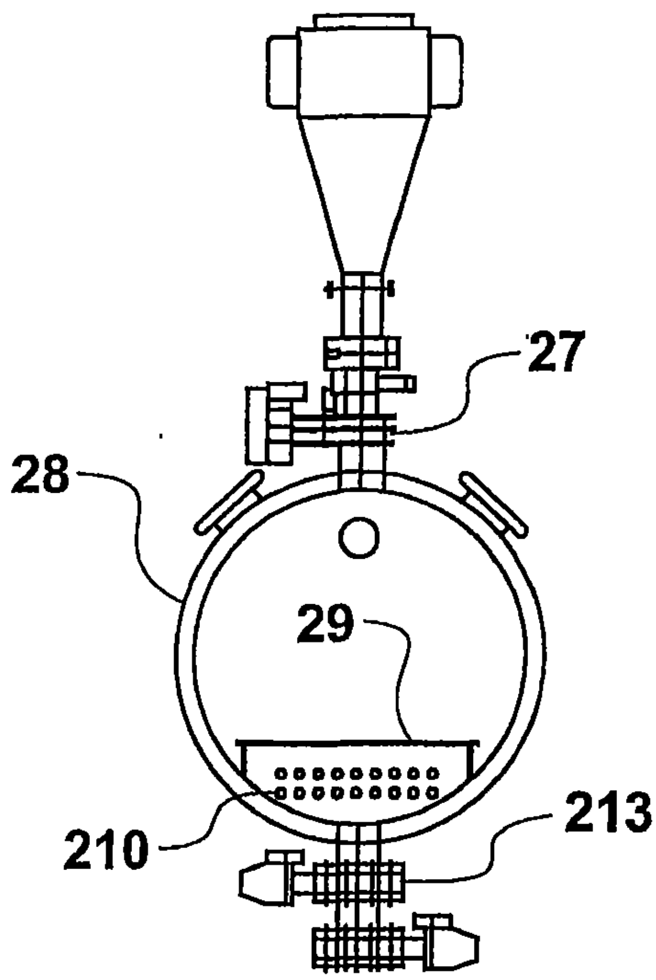

Figura 5

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REIVINDICACIONES

MODIFICADAS

LAS REIVINDICACIONES QUE DEFINEN LA INVENCIÓN SON:

1. Un método de secar o concentrar una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella, el cual incluye los pasos de:
mantener una cámara a una temperatura y presión por debajo del punto triple de la sustancia líquida,
inyectar y/o atomizar la sustancia líquida a la cámara para generar una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),
recoger la porción FLS como una capa en una superficie, y
conducir la porción FLS recogida en la superficie,
en donde la superficie conduce la porción FLS recogida a una velocidad que controla el espesor de la capa de FLS recogida en la superficie.

2. Un método como se reivindica en la reivindicación 1, en donde la velocidad de conducción logra sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

3. Un método como se reivindica en la reivindicación 1 o la reivindicación 2, en donde la FEL se condensa por un dispositivo condensador.

4. Un método como se reivindica en una cualquiera de las reivindicaciones 1 a 3, en donde la FEL se extrae de dicha cámara.

5. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde inyectar y/o atomizar la sustancia líquida incluye atomización para obtener un tamaño de partícula predeterminado de FLS.

6. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la inyección y/o atomización de la sustancia líquida se realiza por una o más boquillas.

7. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la velocidad de conducción de FLS sobre dicha superficie obtiene sustancialmente un espesor de monocapa de la FLS sobre dicha superficie.

8. Un método como se reivindica en la reivindicación 7, en donde la monocapa puede tener el espesor de una capa única del tamaño de partícula predeterminado de la porción FLS.

9. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es sustancialmente 500 μm o más.

10. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es menos de sustancialmente 500 μm.

11. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es menos de sustancialmente 200 μm.

12. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la superficie conduce la porción FLS alejándola de las una o más boquillas.

13. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde el método incluye el paso de exponer la capa recogida de porción FLS a un medio de

calefacción, induciendo sustancialmente en esa forma sublimación y generación de una segunda porción de sustancia líquida evaporada ("porción SEL") y un producto.

14. Un método como se reivindica en la reivindicación 13, en donde la porción SEL se condensa por un dispositivo condensador.

15. Un método como se reivindica en la reivindicación 13 o la reivindicación 14, en donde la porción SEL se extrae de la cámara.

16. Un método como se reivindica en una cualquiera de las reivindicaciones 13 a 15, en donde el producto se extrae de la superficie de conducción por un dispositivo de extracción del producto.

17. Un método como se reivindica en una cualquiera de las reivindicaciones 13 a 16, en donde el producto se extrae de la cámara a través del puerto de salida.

18. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde el método se utiliza para procesar una sustancia fluidizada, o una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella.

19. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, donde la sustancia se selecciona de una o más de las siguientes: una suspensión de café, leche líquida, jugos de frutas y/o vegetales.

20. Un aparato para secar o concentrar una sustancia líquida, el cual comprende:
una cámara y un dispositivo de reducción de presión de la cámara, y uno o más puertos de inyección a través de los cuales se inyecta la

sustancia líquida a la cámara,
una superficie de recolección que recoge una porción de sustancia líquida congelada de la sustancia líquida,
en donde el dispositivo de reducción de presión mantiene la cámara a una presión por debajo de por lo menos la presión de punto triple de la sustancia líquida, para hacer que la sustancia líquida inyectada se separe en una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),
de modo que, en uso, la porción FLS se acumule como una capa en la superficie de recolección y se conduzca lejos de los uno o más puertos de inyección a una velocidad que permita controlar el espesor de la capa de la porción FLS.

21. Un aparato como se reivindica en la reivindicación 20, en donde la velocidad de conducción obtiene sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

22. Un aparato como se reivindica en la reivindicación 20 o la reivindicación 21, en donde la porción FEL se condensa por un dispositivo condensador.

23. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 22, en donde dicha FEL se extrae de dicha cámara.

24. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a la reivindicación 23, en donde el uno o más puertos de inyección comprenden por lo menos una boquilla de aspersión o atomización.

25. Un aparato como se reivindica en la reivindicación 24, en donde la boquilla o boquillas determinan sustancialmente el tamaño de la

porción FLS generada para lograr un tamaño de partícula predeterminado.

26. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 25, en donde el dispositivo de reducción de presión es una bomba de evacuación de gas.

27. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 26, en donde el dispositivo condensador es un serpentín o serpentines refrigerados.

28. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 27, en donde el dispositivo condensador se enfría con un refrigerante.

29. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 28, en donde la superficie de recolección incluye uno, o una combinación, de los siguientes transportadores mecánicos: una configuración de cinta sinfín, una bandeja inclinada para favorecer que la porción de sustancia líquida congelada se deslice desde el uno o más puertos de inyección, una bandeja vibradora.

30. Un aparato como se reivindica en la reivindicación 29, en donde la bandeja inclinada y/o bandeja vibradora incluye una superficie de fricción reducida.

31. Un aparato como se reivindica en la reivindicación 30, en donde la superficie de fricción reducida comprende politetrafluroetileno (PTFE).

32. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 31, en donde la capa de la porción FLS sobre

dicha superficie de recolección se expone a un medio de calefacción, a medida que se conduce lejos del uno o más puertos de inyección.

33. Un aparato como se reivindica en la reivindicación 32, en donde el medio de calefacción sustancialmente induce sublimación de la capa de porción FLS sobre dicha superficie para formar una segunda porción de sustancia líquida evaporada ("SEL") y un producto.

34. Un aparato como se reivindica en la reivindicación 32 o la reivindicación 33, en donde el medio de calefacción es una, o una combinación, de las siguientes fuentes de energía: lámparas infrarrojas, lámparas halógenas, lámparas incandescentes, microondas, o calentamiento óhmico de dicha superficie.

35. Un aparato como se reivindica en la reivindicación 33 o la reivindicación 34, en donde un dispositivo de extracción del producto se emplea para extraer el producto de la superficie de recolección.

36. Un aparato como se reivindica en la reivindicación 35, en donde el dispositivo de extracción del producto es un medio raspador y/o un medio de arrastre de cepillo.

37. Un aparato como se reivindica en la reivindicación 36, en donde el medio raspador contacta sustancialmente la superficie, con el producto sobre ella, y desplaza el producto de la superficie al puerto de salida.

38. Un aparato como se reivindica en la reivindicación 36, en donde el medio de arrastre de cepillo es un cepillo giratorio o cepillo fijo, que contacta sustancialmente la superficie, con el producto sobre

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ella, y dirige el producto de la superficie al puerto de salida.

39. Un aparato como se reivindica en una cualquiera de las reivindicaciones 33 a 37, en donde el producto se extrae a través de un puerto de salida de la cámara.

40. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 39, en donde la cámara se mantiene a una presión de sustancialmente 611.3 Pa o menos.

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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

12 SEP 2006
WIPO PCT

Applicant's or agent's file reference 230677X374	FOR FURTHER ACTION	See Form PCT/IPEA/416
International application No. PCT/NZ2005/000089	International filing date *(day/month/year)* 2 May 2005	Priority date *(day/month/year)* 1 May 2004
International Patent Classification (IPC) or national classification and IPC Int. Cl. ***B01D 1/18*** (2006.01) ***F26B 5/06*** (2006.01)		
Applicant AGRESEARCH LIMITED et al		

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 3 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
   a. [X] *(sent to the applicant and to the International Bureau)* a total of **11** sheets, as follows:
      - [X] sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
      - [ ] sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
   b. [ ] *(sent to the International Bureau only)* a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or table related thereto, in electronic form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).
4. This report contains indications relating to the following items:

[X]	Box No. I	Basis of the report
[ ]	Box No. II	Priority
[ ]	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
[ ]	Box No. IV	Lack of unity of invention
[X]	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
[ ]	Box No. VI	Certain documents cited
[ ]	Box No. VII	Certain defects in the international application
[ ]	Box No. VIII	Certain observations on the international application

Date of submission of the demand	Date of completion of this report
15 November 2005	01 September 2006
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer **Tharu Fernando** Telephone No. (02) 6283 2486

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/NZ2005/000089

Box No. I Basis of the report

1. With regard to the language, this report is based on:

[X] The international application in the language in which it was filed

[ ] A translation of the international application into , which is the language of a translation furnished for the purposes of:

[ ] international search (under Rules 12.3(a) and 23.1 (b))

[ ] publication of the international application (under Rule 12.4(a))

[ ] international preliminary examination (Rules 55.2(a) and/or 55.3(a))

2. With regard to the elements of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

[ ] the international application as originally filed/furnished

[X] the description:

pages 1-21 as originally filed/furnished
pages* received by this Authority on with the letter of
pages* received by this Authority on with the letter of

[X] the claims:

pages as originally filed/furnished
pages* as amended (together with any statement) under Article 19
pages* 22-32 received by this Authority on 23 December 2005 with the letter of 23 December 2005
pages* received by this Authority on with the letter of

[X] the drawings:

pages 1-4 as originally filed/furnished
pages* received by this Authority on with the letter of
pages* received by this Authority on with the letter of

[ ] a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing.

3. [ ] The amendments have resulted in the cancellation of:

[ ] the description, pages
[ ] the claims, Nos.
[ ] the drawings, sheets/figs
[ ] the sequence listing *(specify)*:
[ ] any table(s) related to the sequence listing *(specify)*:

4. [ ] This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

[ ] the description, pages
[ ] the claims, Nos.
[ ] the drawings, sheets/figs
[ ] the sequence listing *(specify)*:
[ ] any table(s) related to the sequence listing *(specify)*:

* *If item 4 applies, some or all of those sheets may be marked "superseded."*

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INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/NZ2005/000089

Box No. V **Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims	1-40	YES
	Claims	41-108	NO
Inventive step (IS)	Claims	1-40	YES
	Claims	41-108	NO
Industrial applicability (IA)	Claims	1-108	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

D3: US 4033048

__Novelty (N) and Inventive Step (IS)__ **(Claims 1-108)**

Claim 41 is directed to a drying process for a liquid substance wherein the liquid substance is atomised, cooled to initiate a phase change, conveyed into a drying chamber under vacuum, heated so as to effect sublimation and then collecting the substantially dried atomised liquid substance from the chamber.

D3 anticipates this claim by disclosing spraying a liquid product (*atomised liquid*) into a freezing chamber, allowing a powder to form (*phase change*) which then falls onto a conveyor surface in a drying chamber. Heat is also applied allowing excess moisture to sublimate (*heated so as to effect sublimation*) and then the dried product is collected in a collection chamber.

Appended claims 42-108 are also anticipated by this document.

According to the attorney's response, the present invention is characterised by controlling the thickness of spray frozen liquid substances. The attorney further indicates that the invention is unlikely to be successful if this aspect were not included, and that this characteristic is essential in allowing the invention to function efficiently and successfully. However, claim 41 is absent on this aspect of the invention and as such is anticipated by D3.

Claims 1-40, which are characterised by the particular characteristic mentioned above, are novel and inventive.

THE CLAIMS DEFINING THE INVENTION ARE:

1. A method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein including the steps of:

holding a chamber at a temperature and pressure below the triple-point of the liquid substance,

injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

collecting the FLS portion as a layer on a surface, and

conveying the collected FLS portion on the surface,

wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface.

2 A method as claimed in claim 1, wherein the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

3. A method as claimed in claim 1 or claim 2, wherein the FEL is condensed by a condensing device.

4. A method as claimed in anyone of claims 1 to 3, wherein the FEL is removed from said chamber.

5. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS.

6. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising of the liquid substance is performed by one or more nozzles.

7. A method as claimed in any one of the preceding claims, wherein the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface.

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8. A method as claimed in claim 7, wherein the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion.

9. A method as claimed in claim 8, wherein the predetermined particle size is substantially 500 µm or more.

10. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 500 µm.

11. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 200 µm.

12. A method as claimed in any one of the preceding claims, wherein the surface conveys the FLS portion away from the one or more nozzles.

13. A method as claimed in any one of the preceding claims, wherein the method includes the step of exposing the collected layer of FLS portion to a heating means thereby substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product.

14. A method as claimed in claim 13, wherein the SEL portion is condensed by a condensing device.

15. A method as claimed in claim 13 or claim 14, wherein the SEL portion is removed from the chamber.

16. A method as claimed in any one of claims 13 to 15, wherein the product is removed from the conveying surface by a product removal device.

17. A method as claimed in any one of claims 13 to 16, wherein the product is removed from the chamber via the outlet port.

18. A method as claimed in any one of the preceding claims, wherein the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein.

19. A method as claimed in any one of the preceding claims where the substance is selected from one or more of the following: a slurry of coffee, liquid milk, fruit and/or vegetable juices.

20. An apparatus for drying or concentrating a liquid substance comprising:

a chamber and a chamber pressure reduction device, and

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one or more injection ports through which the liquid substance is injected to the chamber,

a collection surface which collects a frozen liquid substance portion of the liquid substance,

wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled.

21. An apparatus as claimed in claim 20, wherein the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

22. An apparatus as claimed in claim 20 or claim 21, wherein the FEL portion is condensed by a condensing device.

23. An apparatus as claimed in any one of claims 20 to 22, wherein said FEL is removed from said chamber.

24. An apparatus as claimed in any one claims 20 to claim 23, wherein the one or more injection ports comprises at least one spray or atomisation nozzle.

25. An apparatus as claimed in claim 24, wherein the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle size.

26. An apparatus as claimed in any one of claims 20 to 25, wherein the pressure reduction device is a gas evacuation pump.

27. An apparatus as claimed in any one of claims 20 to 26, wherein the condensing device is a cooled coil or coils.

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28. An apparatus as claimed in any one of claims 20 to 27, wherein the condensing device is cooled with a refrigerant.

29. An apparatus as claimed in any one of claims 20 to 28, wherein the collection surface includes one, or a combination of, the following conveyors: a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray.

30. An apparatus as claimed in claim 29, wherein the angled tray and/or vibrating tray includes a reduced friction surface.

31. An apparatus as claimed in claim 30, wherein the reduced friction surface comprises polytetrafluroethylene (PTFE).

32. An apparatus as claimed in any one of claims 20 to 31, wherein the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports.

33. An apparatus as claimed in claim 32, wherein the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product.

34. An apparatus as claimed in claim 32 or claim 33, wherein the heating means is one or a combination of the following energy sources: infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface.

35. An apparatus as claimed in claim 33 or claim 34, wherein a product removal device is employed to remove the product from the collection surface.

36. An apparatus as claimed in claim 35, wherein the product removal device is a scraping means and/or a brushing means.

37. An apparatus as claimed in claim 36, wherein the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port.

38. An apparatus as claimed in claim 36, wherein the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port.

39. An apparatus as claimed in any one of claims 33 to 37, wherein the product is removed via a chamber outlet port.

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40. An apparatus as claimed in any one of claims 20 to 39, wherein the chamber is held at a pressure of substantially 611.3 Pa or less.

41. A drying process for a liquid substance comprising the steps of:

atomising the liquid substance,

cooling the atomised liquid substance (ALS) to initiate a phase change,

conveying the ALS into a drying chamber under a vacuum,

heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber.

42. A drying process as claimed in claim 41, wherein the liquid substance is chilled from an initial temperature prior to atomising.

43. A drying process as claimed in claim 41 or claim 42, wherein the ALS conveyed into the drying chamber is also conveyed through the drying chamber.

44. A drying process as claimed in any one of claims 41 to 43, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

45. A drying process as claimed in any one of claims 42 to 44, wherein the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature.

46. A drying process as claimed in any one of claims 41 to 45, wherein atomising the liquid substance achieves a predetermined particle size.

47. A drying process as claimed in claim 46, wherein the predetermined particle size is substantially 500 μm or more.

48. A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 500 μm.

49. A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 200 μm.

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50. A drying process as claimed in any one of claims 41 to 49, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

51. A drying process as claimed in any one of claims 42 to 50, wherein chilling is undertaken with direct or indirect contact with a cold fluid.

52. A drying process as claimed in claim 51, wherein said cold fluid is air.

53. A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is substantially 0°C or less.

54. A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is less than substantially -20°C.

55. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a counter current configuration.

56. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a co-current configuration.

57. A drying process as claimed in any one of claims 41 to 56, wherein the ALS is reduced to a temperature below its eutectic temperature.

58. A drying process as claimed in any one of claims 41 to 57, wherein the ALS is conveyed to a separator.

59. A drying process as claimed in any one of claims 41 to 57, wherein said ALS is conveyed pneumatically to a separator.

60. A drying process as claimed in claim 58 or claim 59, wherein said separator is a gas-solid separation device.

61. A drying process as claimed in claim 60, wherein said gas-solid separation device is a cyclone.

62. A drying process as claimed in claim 60 or claim 61, wherein the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step.

63. A drying process as claimed in claim 61 or claim 62, wherein one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet.

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64. A drying process as claimed in any one of claims 60 to 63, wherein separated solids from said gas-solid separation device enter a vacuumous drying chamber.

65. A drying process as claimed in any one of claims 41 to 64, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device.

66. A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

67. A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

68. A drying process as claimed in any one of claims 41 to 67, wherein said ALS is conveyed via a surface.

69. A drying process as claimed in any one of claims 41 to 68, wherein said ALS is conveyed via a vibrating surface.

70. A drying process as claimed in claim 69, wherein said vibrating surface is a vibrating tray.

71. A drying process as claimed in claim 69 or claim 70, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

72. A drying process as claimed in claim 70 or claim 71, wherein said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation.

73. A drying process as claimed in any one of claims 41 to 72, wherein a vapour produced by sublimation is removed from the drying chamber.

74. A drying process as claimed in claim 73, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers.

75. A drying process as claimed in any one of claims 41 to 74, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

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76. A drying process as claimed in any one of claims 41 to 75, wherein said temperature gradient is provided by an energy source.

77. A drying process as claimed in claim 76, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

78. A drying process as claimed in any one of claims 41 to 77, wherein atomising the liquid substance achieves a predetermined particle size.

79. A drying process as claimed in claim 78, wherein the predetermined particle size is substantially 500 µm or more.

80. A drying process as claimed in claim 78, wherein the predetermined particle size is less than substantially 500 µm.

81. A drying process as claim in claim 78, wherein the predetermined particle size is less than substantially 200 µm.

82. A drying process as claimed in any one of claims 60 to 81, wherein a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber.

83. A drying process as claimed in any one of claims 41 to 82, wherein a product produced is substantially reduced is free of liquid compared to the liquid substance.

84. An apparatus for a drying process for a liquid substance comprising:

an atomiser capable of atomising a liquid substance,

a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,

a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,

an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and

a collector capable of collecting the dried ALS.

85. An apparatus for a drying a drying process as claimed in claim 84, wherein a

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chiller capable of chilling a liquid substance from an initial temperature to a lower temperature is provided prior to the liquid substance atomiser.

86. An apparatus for a drying a drying process as claimed in claim 84 or claim 85, wherein the apparatus includes a conveyor capable of conveying the ALS through the drying chamber.

87. An apparatus for a drying a drying process as claimed in any one of claims 84 to 86, wherein the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation.

88. An apparatus for a drying process as claimed in claim 87, wherein the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber.

89. An apparatus for a drying process as claimed in any one of claims 84 to 88, wherein atomising the liquid substance achieves a predetermined particle size.

90. An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is substantially 500 μm or more.

91. An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is less than substantially 500 μm.

92. An apparatus for a drying process as claimed in claim 89, wherein the predetermined size is less than substantially 200 μm.

93. An apparatus for a drying process as claimed in any one of claims 84 to 92, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

94. An apparatus for a drying process as claimed in claim 93, wherein said cold gas is air.

95. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is substantially 0°C or less.

96. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is less than substantially -20°C.

97. An apparatus for a drying process as claimed in any one of claims 84 to 96, wherein the vacuum of said vacuumous drying chamber is created by a

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pressure reduction device.

98. An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

99. An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

100. An apparatus for a drying process as claimed in any one of claims 84 to 99, wherein said ALS is conveyed via a vibrating surface.

101. An apparatus for a drying process as claimed in claim 100, wherein said vibrating surface is a vibrating tray.

102. An apparatus for a drying process as claimed in claim 101, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

103. An apparatus for a drying process as claimed in claim 84 or claim 102, wherein ALS is conveyed through a temperature gradient to substantially effect sublimation.

104. An apparatus for a drying process as claimed in claim 84 or claim 103, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers.

105. An apparatus for a drying process as claimed in claim 103, wherein said temperature gradient is provided by an energy source.

106. An apparatus for a drying process as claimed in claim 105, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

107. A drying chamber for use with the apparatus as claimed in any one of claims 20 to 40 which comprises:

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray.

108. A drying chamber for use with the apparatus as claimed in anyone of claims 64 to 106 which comprises:

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray.

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ABSTRACT

This invention relates to a process and/or method and associated apparatus for use in spray freeze drying a fluid substance such as fruit juice, pharmaceuticals, nutraceuticals, tea and coffee. A fluid substance is sprayed (atomised) and reduced in temperature to below its eutectic temperature; thereby inducing a phase change of fluids in the fluid substance. The atomised fluid substance (AFS) is then subjected to a vacuumous drying chamber in which the AFS is subjected to an energy source to assist sublimation of fluid from the AFS as it is conveyed through the chamber. Such an energy source can provide a temperature gradient through which the AFS passes and is exposed to, all carried out so as to preferably minimise or reduce the likelihood of degradation of the substance being dried (reducing the fluid content).

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TRATADO DE COOPERACIÓN EN MATERIA DE PATENTES

PCT

INFORME DE EXAMEN PRELIMINAR INTERNACIONAL

(Capítulo II del Tratado de Cooperación en Matena de Patentes)

(Artículo 36 y Regla 70 PCT)

No de Expediente del Solicitante o Apoderado 230677X374	PROCEDIMIENTO POSTERIOR	Véase formulano PCT/IPEA/416
No Solicitud Internacional PCT/NZ2005/000089	Fecha Internacional de Presentación *(día/mes/año)* 2 de Mayo de 2005	Fecha de Prioridad *(día/mes/año)* 1 de Mayo de 2004
Clasificación Internacional de Patente (IPC) o Clasificación Nacional e IPC Int Cl ***B01D 1/18*** (2006.01) ***F26B 5/06*** (2006 01)		
Solicitante AGRESEARCH LIMITED et al		

1. El presente informe es el informe de examen preliminar internacional, establecido por esta Oficina Encargada del Examen Preliminar Internacional bajo Artículo 35 y transmitido a solicitante de acuerdo con el Artículo 36
2. El presente INFORME comprende un total de 3 hojas, inclusive esta portada
3. Este importe se acompaña igualmente por ANEXOS, que comprenden
   - a ☒ *(enviado al solicitante y a la Oficina Internacional)* un total de 11 hojas, como sigue
     - ☒ hojas de la descripción, reivindicaciones y/o dibujos que se han corregido y constituyen la base de el presente informe y/o hojas que contienen rectificaciones autorizadas por esta Oficina (véase Regla 70 16 y Sección 607 de las Instrucciones Administrativas)
     - ☐ hojas que reemplazan hojas anteriores, pero que esta Oficina considera que contienen una corrección que va más allá de la divulgación en la solicitud internacional tal como se presentó, como se indica en el Item 4 de Cuadro No I y el Cuadro Adicional
   - b ☐ (enviado a la Oficina Internacional únicamente) un total de (indicar tipo y número de medios electrónicos)) ,que contiene un listado de secuencias y/o tablas relacionadas con este asunto, en formato de lectura por computador únicamente, como se indica en el Cuadro Adicional Relativo al Listado de Secuencias (véase Sección 802 de las Instrucciones Administrativas)

4. El presente informe contiene datos con respecto a los siguientes puntos
   - ☒ Cuadro No I — Base del Informe
   - ☐ Cuadro No II — Prioridad
   - ☐ Cuadro No III — Sin elaboración de un dictamen sobre novedad, actividad inventiva y posibilidad de aplicación industrial
   - ☐ Cuadro No IV — Uniformidad deficiente de la invención
   - ☒ Cuadro No V — Declaración justificada según Artículo 35(2) con respecto a la novedad, la actividad inventiva y la posibilidad de aplicación industrial, documentos y explicaciones que sustentan esta declaración
   - ☐ Cuadro No VI — Ciertos documentos enumerados
   - ☐ Cuadro No VII — Ciertas deficiencias del registro internacional
   - ☐ Cuadro No VIII — Ciertas observaciones relacionadas con la solicitud internacional

Fecha de presentación de la petición	Fecha de la preparación de el presente informe
15 de noviembre de 2005	1 de septiembre de 2006
Nombre y dirección postal de la Oficina Encargada del Examen Preliminar Internacional AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA Dirección e-mail pct@ipaustralia gov au Fax No (02) 6285 3929	Empleado Autorizado Tharu Fernando Teléfono No (02) 6283 2486

Formulario PCT/IPEA/409 (portada) (Abril 2005)

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INFORME PRELIMINAR INTERNACIONAL SOBRE PATENTABILIDAD

Solicitud Internacional No
PCT/NZ2005/000089

Cuadro No. I Base del Informe

1. Con respecto al **idioma:** El presente informe se basa en

☒ La solicitud internacional en el idioma en el cual se radicó

☐ Una traducción de la solicitud internacional al , que es el idioma de una traducción suministrada para los efectos de

- ☐ la búsqueda internacional (según las reglas 12.3 y 23 1 (b))
- ☐ la publicación de la solicitud internacional (según la regla 12 4)
- ☐ el examen preliminar internacional (según las reglas 55 2 y/o 55 3)

2 Con respecto a los **elementos** de la solicitud internacional, este informe se basa en *(hojas intercambiables que se han suministrado a la oficina receptora en respuesta a una invitación bajo Artículo 14 se tienen en este informe por "presentadas originalmente" y no se anexan a este informe).*

☐ la solicitud internacional tal como se radicó/entregó originalmente

☒ la descripción

páginas **1-21** como se radicaron/entregaron originalmente

páginas* recibidas por esta Oficina el con la carta de

páginas* recibidas por esta Oficina el con la carta de

☒ las reivindicaciones

páginas como se radicaron/entregaron originalmente

páginas como se modificaron (junto con cualquier declaración) bajo artículo 19

páginas **22-32** recibidas por esta Oficina el **23 de diciembre de 2005** con la carta de **23 de diciembre de 2005**

páginas* recibidas por esta Oficina el con la carta de

☒ los dibujos

páginas **1-4** como se radicaron/entregaron originalmente

páginas* recibidas por esta Oficina el con la carta de

páginas* recibidas por esta Oficina el con la carta de

☐ Un listado de secuencias y/o cualquier tabla(s) relacionada(s)– véase Cuadro Adicional relativo al Listado de Secuencias

3 ☐ Las correcciones han resultado en la cancelación de

- ☐ la descripción, páginas
- ☐ las reivindicaciones, Nos
- ☐ los dibujos, hojas/figs.
- ☐ el listado de secuencias *(especificar)*
- ☐ cualquier tabla(s) relacionada con el listado de secuencias *(especificar)*

4 ☐ El presente informe se preparó como si no se hubieran hecho (algunas de) las correcciones anexadas a este informe y enumeradas a continuación ya que éstas, por las razones aducidas, en concepto de la oficina van más allá del contenido de divulgación en la versión presentada originalmente (Regla 70.2 (c)).

- ☐ la descripción, páginas
- ☐ las reivindicaciones, Nos
- ☐ los dibujos, hojas/figs
- ☐ el listado de secuencias *(especificar)*
- ☐ cualquier tabla(s) relacionada con el listado de secuencias *(especificar)*

* *Si se aplica el punto 4, algunas o todas estas hojas pueden estar marcadas como "reemplazadas "*

INFORME PRELIMINAR INTERNACIONAL SOBRE PATENTABILIDAD

Solicitud Internacional No
PCT/NZ2005/000089

Cuadro No. V Declaración motivada bajo Artículo 35(2) en cuanto a la novedad, la actividad inventiva y la posibilidad de aplicación industrial; documentos y explicaciones que sustentan esta declaración

1. DECLARACIÓN

Novedad (N)	Reivindicaciones	**1-40**	SI
	Reivindicaciones	**41-108**	NO
Actividad Inventiva (IS)	Reivindicaciones	**1-40**	SI
	Reivindicaciones	**41-108**	NO
Posibilidad de Aplicación Industrial (IA)	Reivindicaciones	**1-108**	SI
	Reivindicaciones		NO

2 Documentos y explicaciones (Regla 70 7)

D3: US 4033048

<u>Novedad (N) y Actividad Inventiva (IS)</u> (Reivindicaciones 1-108)
La reivindicación 41 se dirige a un proceso de secado para una sustancia líquida, en donde la sustancia líquida se atomiza, se enfría para iniciar un cambio de fase, se conduce a una cámara de secado bajo vacío, se calienta con el fin de efectuar sublimación y luego recolectar de la cámara la sustancia líquida atomizada sustancialmente seca.

D3 anticipa esta reivindicación que divulga atomizar un producto líquido (*líquido atomizado*) a una cámara de congelamiento, permitiendo que se forme un polvo (*cambio de fase*), el cual cae entonces sobre una superficie de un transportador mecánico en una cámara de secado. Se aplica también calor, lo cual permite sublimar el exceso de humedad (*se calienta con el fin de efectuar sublimación*) y luego el producto seco se recoge en una cámara de recolección.

En consecuencia, las reivindicaciones adjuntas 42-108 son anticipadas por este documento.

Según la respuesta del apoderado, la presente invención se caracteriza por controlar el espesor de las sustancias líquidas congeladas por atomización. El apoderado indica además que la invención no tiene probabilidades de éxito si no se incluyera este aspecto, y que esta característica es esencial para permitir que la invención funcione eficiente y exitosamente. No obstante, la reivindicación 41 está ausente en este aspecto de la invención y, como tal, se anticipa por **D3**.

Las reivindicaciones 1-40, que se caracterizan por la característica particular ya mencionada, son novedosas e inventivas.

LAS REIVINDICACIONES QUE DEFINEN LA INVENCIÓN SON:

1. Un método de secar o concentrar una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella, el cual incluye los pasos de:
   mantener una cámara a una temperatura y presión por debajo del punto triple de la sustancia líquida,
   inyectar y/o atomizar la sustancia líquida a la cámara para generar una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),
   recoger la porción FLS como una capa en una superficie, y
   conducir la porción FLS recogida en la superficie,
   en donde la superficie conduce la porción FLS recogida a una velocidad que controla el espesor de la capa de FLS recogida en la superficie.

2. Un método como se reivindica en la reivindicación 1, en donde la velocidad de conducción logra sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

3. Un método como se reivindica en la reivindicación 1 o la reivindicación 2, en donde la FEL se condensa por un dispositivo condensador.

4. Un método como se reivindica en una cualquiera de las reivindicaciones 1 a 3, en donde la FEL se extrae de dicha cámara.

5. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde inyectar y/o atomizar la sustancia líquida incluye atomización para obtener un tamaño de partícula predeterminado de FLS.

6. Un método como se reivindica en una cualquiera de las

reivindicaciones precedentes, en donde la inyección y/o atomización de la sustancia líquida se realiza por una o más boquillas.

7. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la velocidad de conducción de FLS sobre dicha superficie obtiene sustancialmente un espesor de monocapa de la FLS sobre dicha superficie.

8. Un método como se reivindica en la reivindicación 7, en donde la monocapa puede tener el espesor de una capa única del tamaño de partícula predeterminado de la porción FLS.

9. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es sustancialmente 500 μm o más.

10. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es menos de sustancialmente 500 μm.

11. Un método como se reivindica en la reivindicación 8, en donde el tamaño de partícula predeterminado es menos de sustancialmente 200 μm.

12. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde la superficie conduce la porción FLS alejándola de las una o más boquillas.

13. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde el método incluye el paso de exponer la capa recogida de porción FLS a un medio de calefacción, induciendo sustancialmente en esa forma sublimación y generación de una segunda porción de sustancia líquida evaporada ("porción SEL") y

un producto.

14. Un método como se reivindica en la reivindicación 13, en donde la porción SEL se condensa por un dispositivo condensador.

15. Un método como se reivindica en la reivindicación 13 o la reivindicación 14, en donde la porción SEL se extrae de la cámara.

16. Un método como se reivindica en una cualquiera de las reivindicaciones 13 a 15, en donde el producto se extrae de la superficie de conducción por un dispositivo de extracción del producto.

17. Un método como se reivindica en una cualquiera de las reivindicaciones 13 a 16, en donde el producto se extrae de la cámara a través del puerto de salida.

18. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde el método se utiliza para procesar una sustancia fluidizada, o una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella.

19. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, donde la sustancia se selecciona de una o más de las siguientes: una suspensión de café, leche líquida, jugos de frutas y/o vegetales.

20. Un aparato para secar o concentrar una sustancia líquida, el cual comprende:

una cámara y un dispositivo de reducción de presión de la cámara, y uno o más puertos de inyección a través de los cuales se inyecta la sustancia líquida a la cámara,

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una superficie de recolección que recoge una porción de sustancia líquida congelada de la sustancia líquida,

en donde el dispositivo de reducción de presión mantiene la cámara a una presión por debajo de por lo menos la presión de punto triple de la sustancia líquida, para hacer que la sustancia líquida inyectada se separe en una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),

de modo que, en uso, la porción FLS se acumule como una capa en la superficie de recolección y se conduzca lejos de los uno o más puertos de inyección a una velocidad que permita controlar el espesor de la capa de la porción FLS.

21. Un aparato como se reivindica en la reivindicación 20, en donde la velocidad de conducción obtiene sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

22. Un aparato como se reivindica en la reivindicación 20 o la reivindicación 21, en donde la porción FEL se condensa por un dispositivo condensador.

23. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 22, en donde dicha FEL se extrae de dicha cámara.

24. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a la reivindicación 23, en donde el uno o más puertos de inyección comprenden por lo menos una boquilla de aspersión o atomización.

25. Un aparato como se reivindica en la reivindicación 24, en donde la boquilla o boquillas determinan sustancialmente el tamaño de la

porción FLS generada para lograr un tamaño de partícula predeterminado.

26. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 25, en donde el dispositivo de reducción de presión es una bomba de evacuación de gas.

27. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 26, en donde el dispositivo condensador es un serpentín o serpentines refrigerados.

28. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 27, en donde el dispositivo condensador se enfría con un refrigerante.

29. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 28, en donde la superficie de recolección incluye uno, o una combinación, de los siguientes transportadores mecánicos: una configuración de cinta sinfín, u na bandeja inclinada para favorecer que la porción de sustancia líquida congelada se deslice desde el uno o más puertos de inyección, una bandeja vibradora.

30. Un aparato como se reivindica en la reivindicación 29, en donde la bandeja inclinada y/o bandeja vibradora incluye una superficie de fricción reducida.

31. Un aparato como se reivindica en la reivindicación 30, en donde la superficie de fricción reducida comprende politetrafluroetileno (PTFE).

32. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 31, en donde la capa de la porción FLS sobre

dicha superficie de recolección se expone a un medio de calefacción, a medida que se conduce lejos del uno o más puertos de inyección.

33. Un aparato como se reivindica en la reivindicación 32, en donde el medio de calefacción sustancialmente induce sublimación de la capa de porción FLS sobre dicha superficie para formar una segunda porción de sustancia líquida evaporada ("SEL") y un producto.

34. Un aparato como se reivindica en la reivindicación 32 o la reivindicación 33, en donde el medio de calefacción es una, o una combinación, de las siguientes fuentes de energía: lámparas infrarrojas, lámparas halógenas, lámparas incandescentes, microondas, o calentamiento óhmico de dicha superficie.

35. Un aparato como se reivindica en la reivindicación 33 o la reivindicación 34, en donde un dispositivo de extracción del producto se emplea para extraer el producto de la superficie de recolección.

36. Un aparato como se reivindica en la reivindicación 35, en donde el dispositivo de extracción del producto es un medio raspador y/o un medio de arrastre de cepillo.

37. Un aparato como se reivindica en la reivindicación 36, en donde el medio raspador contacta sustancialmente la superficie, con el producto sobre ella, y desplaza el producto de la superficie al puerto de salida.

38. Un aparato como se reivindica en la reivindicación 36, en donde el medio de arrastre de cepillo es un cepillo giratorio o cepillo fijo, que contacta sustancialmente la superficie, con el producto sobre ella, y dirige el producto de la superficie al puerto de salida.

39. Un aparato como se reivindica en una cualquiera de las

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reivindicaciones 33 a 37, en donde el producto se extrae a través de un puerto de salida de la cámara.

40. Un aparato como se reivindica en una cualquiera de las reivindicaciones 20 a 39, en donde la cámara se mantiene a una presión de sustancialmente 611.3 Pa o menos.

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XX

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 230677X374	FOR FURTHER ACTION	see Form PCT/ISA/220 as well as, where applicable, item 5 below.
International application No. PCT/NZ2005/000089	International filing date *(day/month/year)* 2 May 2005	(Earliest) Priority Date *(day/month/year)* 1 May 2004
Applicant AGRESEARCH LIMITED et al		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 6 sheets.

☐ It is also accompanied by a copy of each prior art document cited in this report

1. **Basis of the report**

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ The international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23 1(b))

b. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2 ☐ **Certain claims were found unsearchable** (See Box No. II).

3 ☒ **Unity of invention is lacking** (See Box No III).

4 With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6 With regard to the **drawings**,

a. the figure of the drawings to be published with the abstract is Figure No. 1

☒ as suggested by the applicant.

☐ as selected by this Authority, because the applicant failed to suggest a figure.

☐ as selected by this Authority, because this figure better characterizes the invention

b. ☐ none of the figures is to be published with the abstract

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/NZ2005/000089

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
   because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
   because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
   because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See supplemental sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims
2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims, it is covered by claims Nos.:

Remark on Protest ☐ The additional search fees were accompanied by the applicant's protest

☐ No protest accompanied the payment of additional search fees.

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/NZ2005/000089

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. 7: B01D 1/18; F26B 5/06

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
DWPI: A23L 3/44, B01D 1/16, 1/18, 1/20, 9/04, F26B 5/06, 17/10; & Keywords - spray+, inject+, atomis+, atomiz+, freez+, froz+, dry+, concentrat+, (triple point)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	Derwent Abstract Accession No. 2004-576721/56, Class D13, J08, JP 2004232883 A (ULVAC CORP) 19 August 2004 Abstract	1-106
P, X	US 2004/0154317 A (SHEKUNOV ET AL) 12 August 2004 Whole document	1-106
X	Derwent Abstract Accession No. 2004-094584/10, Class D14 JP 2004012015 A (OKAWARA SEISAKUSHO KK) 15 January 2004 Abstract	1-106
X	Derwent Abstract Accession No. 88-358218/50, Class Q76 JP 63269969 A (HOUSE SHOKUHIN KOGYO) 8 November 1988 Abstract	1-106

[X] Further documents are listed in the continuation of Box C [X] See patent family annex

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 August 2005	25 AUG 2005

Name and mailing address of the ISA/AU	Authorized officer
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No (02) 6285 3929	Tharu Fernando Telephone No: (02) 6283 2486

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/NZ2005/000089

C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4033048 A (VAN IKE) 5 July 1977 Whole document	1-106
X	Derwent Abstract Accession No. 82-41035e/20, Class J08 , X25, Q76 SU 0848932 A (PRESERVES FOOD IND) 23 July 1981 Abstract	107
A	US 4810274 A (CHENG ET AL) 7 March 1989 Whole document	1-106

INTERNATIONAL SEARCH REPORT

International application No.
PCT/NZ2005/000089

Supplemental Box
(To be used when the space in any of Boxes I to VIII is not sufficient)

Continuation of Box No III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

The international application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept. In coming to this conclusion the International Searching Authority has found that there are different inventions as follows:

1. Claims 1 to 106 are directed to a method of drying or concentrating a liquid substance whereby the liquid is sprayed into a chamber that is held at a temperature and pressure below the triple-point of the liquid, such that the sprayed liquid generates a frozen liquid substance and an evaporated liquid, the frozen liquid portion is collected and dried to sublime excess moisture and obtain dried solid particles. It is considered that holding the chamber at a temperature and pressure below the triple point of the liquid substance to such that frozen droplets are obtained comprises a first "special technical feature".

2. Claim 107 is directed to a drying chamber comprising a device capable of vibrating a tray, a device capable of reducing the pressure of the chamber, an outlet port and a heat source adapted to act on the tray. It is considered that the drying chamber as a whole comprises a second special technical feature.

Since the abovementioned groups of claims do not share any of the technical features identified, a "technical relationship" between the inventions, as defined in PCT rule 13.2 does not exist. Accordingly the international application does not relate to one invention or to a single inventive concept, a priori.

(4D)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/NZ2005/000089

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member	
JP	2004232883	NONE	
US	2004154317	WO	2004071410
JP	2004012015	NONE	
JP	63269969	NONE	? ordered.
US	4033048	NONE	
SU	0848932	NONE	?
US	4810274	WO	9010838

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX

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PATENT COOPERATION TREATY

From the:
INTERNATIONAL SEARCHING AUTHORITY

To:

Cable, Andy
Private Bag 11907,
Level 9
56 Cawley Street
Ellerslie
Auckland, New Zealand 1005

PCT

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

(PCT Rule 43*bis*.1)

Date of mailing *(day/month/year)* 25 AUG 2005		
Applicant's or agent's file reference 230677X374	FOR FURTHER ACTION See paragraph 2 below	
International application No. PCT/NZ2005/000089	International filing date *(day/month/year)* 2 May 2005	Priority date *(day/month/year)* 1 May 2004
International Patent Classification (IPC) or both national classification and IPC Int. Cl. [7] B01D 1/18; F26B 5/06		
Applicant AGRESEARCH LIMITED et al		

1. This opinion contains indications relating to the following items:

- [X] Box No. I — Basis of the opinion
- [ ] Box No II — Priority
- [ ] Box No. III — Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- [X] Box No IV — Lack of unity of invention
- [X] Box No V — Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- [X] Box No VI — Certain documents cited
- [ ] Box No. VII — Certain defects in the international application
- [X] Box No VIII — Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1*bis*(b) that written opinions of this International Searching Authority will not be so considered

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later

For further options, see Form PCT/ISA/220

3 For further details, see notes to Form PCT/ISA/220.

Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia gov au Facsimile No (02) 6285 3929	Authorized Officer **THARU FERNANDO** Telephone No (02) 6283 2486

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WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.

PCT/NZ2005/000089

Box No. I Basis of the opinion

1. With regard to the language, this opinion has been established on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

   ☐ This opinion has been established on the basis of a translation from the original language into the following language , which is the language of a translation furnished for the purposes of international search (under Rules 12.3 and 23.1(b)).

2. With regard to any nucleotide and/or amino acid sequence disclosed in the international application and necessary to the claimed invention, this opinion has been established on the basis of:

   a. type of material

      ☐ a sequence listing

      ☐ table(s) related to the sequence listing

   b. format of material

      ☐ in written format

      ☐ in computer readable form

   c. time of filing/furnishing

      ☐ contained in the international application as filed.

      ☐ filed together with the international application in computer readable form.

      ☐ furnished subsequently to this Authority for the purposes of search.

3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4 Additional comments:

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WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.

PCT/NZ2005/000089

Box No. IV Lack of unity of invention

1. ☐ In response to the invitation (Form PCT/ISA/206) to pay additional fees the applicant has:

   ☐ paid additional fees

   ☐ paid additional fees under protest

   ☐ not paid additional fees

2. ☒ This Authority found that the requirement of unity of invention is not complied with and chose not to invite the applicant to pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rule 13 1, 13 2 and 13 3 is

   ☐ complied with

   ☒ not complied with for the following reasons:

The international application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept. In coming to this conclusion the International Searching Authority has found that there are different inventions as follows:

1. Claims 1 to 106 are directed to a method of drying or concentrating a liquid substance whereby the liquid is sprayed into a chamber that is held at a temperature and pressure below the triple-point of the liquid, such that the sprayed liquid generates a frozen liquid substance and an evaporated liquid, the frozen liquid portion is collected and dried to sublime excess moisture and obtain dried solid particles. It is considered that holding the chamber at a temperature and pressure below the triple point of the liquid substance to such that frozen droplets are obtained comprises a first "special technical feature".

2. Claim 107 is directed to a drying chamber comprising a device capable of vibrating a tray, a device capable of reducing the pressure of the chamber, an outlet port and a heat source adapted to act on the tray. It is considered that the drying chamber as a whole comprises a second special technical feature

Since the abovementioned groups of claims do not share any of the technical features identified, a "technical relationship" between the inventions, as defined in PCT rule 13.2 does not exist Accordingly the international application does not relate to one invention or to a single inventive concept, a priori.

4. Consequently, this opinion has been established in respect of the following parts of the international application:

   ☒ all parts

   ☐ the parts relating to claims Nos.

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WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY	International application No. PCT/NZ2005/000089

Box No. V **Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims	YES
	Claims 1-107	NO
Inventive step (IS)	Claims	YES
	Claims 1-107	NO
Industrial applicability (IA)	Claims 1-107	YES
	Claims	NO

2. Citations and explanations:

Novelty and Inventive Step (claims 1-107)

The present invention relates to a process and/or method and associated apparatus for use in spray-freeze drying a liquid substance. The liquid substance is sprayed into a chamber at a temperature below its eutectic temperature such that it forms a frozen liquid part and an evaporated liquid part. The frozen particles are conveyed to a dryer under vacuum to remove excess moisture through sublimation thereby providing a dried concentrate.

The closest prior art documents to the present invention include:

D1 JP 2004012015

D2 JP 63269969

D3 US 4033048 A

D4 SU 0848932

D1-D3 describe features defined in claims 1-106. Each citation discloses a process whereby a liquid substance is atomised into a chamber which is at a pressure and temperature such that the liquid droplets divide into a frozen portions and an evaporated portion, after which the frozen portion is dried in a vacuum chamber such that excess moisture is sublimated. Therefore claims 1-106 are disclosed and lack novelty and inventive step.

D4 teaches a vacuum dryer comprising vibrating electrically heated shelves. The figure further discloses a material outlet port as part of the chamber. As such claim 107, which includes the features of a device capable of vibrating a tray, a device for reducing the pressure in the chamber, an outlet port and a heat source for the tray, is anticipated, lacking novelty and inventive step.

Industrial Applicability (claims 1-107)

The invention defined in claims 1-107 is industrially applicable.

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WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.

PCT/NZ2005/000089

Box No. VI Certain documents cited

1. Certain published documents (Rules 43*bis*.1 and 70.10)

	Application No. Patent No.	Publication date *(day/month/year)*	Filing date *(day/month/year)*	Priority date (valid claim) *(day/month/year)*
P,X	JP 2004232883	19 August 2004	28 January 2003	28 January 2003
P,X	US 2004/0154317	12 August 2004	8 May 2003	7 February 2003

JP 2004232883 discloses a spraying-type freeze-drying apparatus that sprays concentrated liquid to a freeze-drying tower through a nozzle, such that the liquid is sprayed as micro freeze material and collected in a collection board, after which it is dried under vacuum and excess moisture sublimed.

US 2004/0154317 teaches a method of freeze-drying whereby a solution is atomised into a chamber and is collected as frozen droplets, after which the frozen solvent is sublimated from the mixture of solid particles leaving behind dried solid particles.

2. Non-written disclosures (Rules 43*bis*.1 and 70 9)

Kind of non-written disclosure	Date of non-written disclosure *(day/month/year)*	Date of written disclosure referring to non-written disclosure *(day/month/year)*

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WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/NZ2005/000089

`ox No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

1. Claim 107 does not define the invention described because it omits features describing the spray freeze-drying process, which from reading the specification as a whole, appear to be essential to the invention.

X02.362

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INTELLECTUAL PROPERTY

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Re. AGRESEARCH LIMITED
PCT Application No. PCT/NZ2005/000089
DRYING PROCESS AND APPARATUS
Our Ref: 230677/60 LV

We refer to the International Search Report of 25 August 2005 and matters arising

SUMMARY

- The cited documents do not disclose the present invention. The present application is both novel and inventive.
- The applicant has amended claim 107 to be dependent on claim 1. This overcomes the examiner's unity of invention objection
- Document 4, SU 0848932, is no longer relevant because of the amendment to claim 107.

PRESENT APPLICATION

The present application is for a method of drying or concentrating a liquid substance having solid particles in suspension including the steps of.

- Holding a chamber at a temperature and pressure below the triple point of the liquid substance; and
- Injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion and a first evaporated liquid substance portion; and

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- Collecting the frozen liquid substance portion as a layer on the surface; and
- Conveying the collected frozen liquid substance portion on the surface; and
- Wherein the surface conveys the collected frozen liquid substance portion at a rate which controls the thickness of the layer of collected frozen liquid substance on the surface.

The key inventive aspect of this invention is that it controls the rate of thickness of product formed on the drying surface This is essential to improve the efficiency of the spray freeze drying process and allows the invention to operate successfully.

None of the cited documents disclose, either alone or in combination, a method of drying which controls the thickness of the layer formed by spray-freezing a substance.

A discussion of each patent follows

Document 1 - Japanese Patent No. 2004012015A.

This patent discloses a drying chamber which is connected to a vacuum generated for drying liquid material

This patent uses a drying zone where the liquid material is sprayed onto a freezing sheet. This freezing sheet is formed from a heat insulating material with a small thermal capacity.

This patent does not even disclose a method of <u>spray</u> freeze drying. Freezing is achieved by the heat insulting material. It is clearly not relevant to the present invention.

Document 2 - Japanese Patent No. 2004232883A.

This discloses a spray freeze drying apparatus where a liquid is sprayed to form a frozen material, the frozen material is collected on a board and subsequently dried under vacuum. The collection board is rotated around a shaft to collect the frozen material.

The invention operates in a tower with the nozzle being located above the collection board The collection board rotates about the central shaft of the tower to collect the powder at the bottom of the tower.

This patent makes no mention of controlling the thickness of frozen product to improve the quality and efficiency of the drying process, and clearly does not disclose the present invention.

( )

Document 3 - United States Patent No. 2004/0154317

This patent discloses a method and device for producing particles. Specifically, the device includes:

- A solution source and a fluid source supplying a compressed fluid (the fluid);
- A mixer in communication with the sources for mixing the solution and the fluid;
- An expansion assembly for spraying the mixture across a pressure drop;
- A freeze dry apparatus
- In the body of the specification the term "compressed fluid" is defined as:

  "a gas that is in a liquid, compressed or supercritical state".

The method disclosed in this invention includes the steps of:

- Mixing a solution and a compressed fluid together;
- Spraying the mixture across a pressure drop into an expansion chamber to effect freezing of the mixture;
- Freeze drying the frozen droplets of solution

There is no reference through the specification to the mechanics of the freeze drying process. It is focused on the step of and means to mix the solution with the compressed fluid.

There is not mention of controlling the thickness of frozen product to improve the quality and efficiency of the spray-freeze drying process.

Further, this patent is directed toward the production of highly porous, flaky substances suitable for drug delivery. The step of mixing the product with the fluid would be unnecessary and undesirable for use in spray freeze drying technology for the food industry

This patent clearly does not anticipate the present invention.

Document 4 – US 4033048A

There are four clear differences between the invention disclosed in the present application and that disclosed in US Patent No. 4,033,048 These are.

- The use of a collection funnel versus direct delivery of frozen product to a transportation means.

US Patent No. 4,033,048 uses a collection funnel to place or position the spray frozen material onto a transportation means (conveyor belt). This is not ideal for two reasons, these being:

- Frozen liquids and in particular those formed from spraying a liquid into a sub triple point vacuum, tend to cake very easily. It is difficult to ensure that the funnel would not block with product after even a short period of operation.

- The funnel with its circular cross-section could not distribute the product evenly across the width of the belt. This means that the thickness of the product layer and hence the required drying times would not be uniform across the belt. This would result in a wide variety of product quality and hence results produced by this invention would be unsatisfactory.

The present application delivers the frozen product directly to the transportation means. This is either via a vibrating tray or a sweeping nozzle that traverses the transportation means.

The present invention produces higher quality results by ensuring a uniform thickness of product is achieved over the drying surface which allows uniform drying times and produces a higher quality product.

- The use of multiple conveyor belts versus single conveyor belts.

The invention described by US Patent No. 4033,048 has multiple conveyor belts. The frozen product would tend to adhere to the drying surface and it would not necessarily fall from the belt under the influence of gravity.

In addition, the patent specification also discloses the use of brushes or scrapers of some form to transfer product to lower belts. This will lead to the loss of product which increases with the number of transfers between belts involved.

These problems do not occur with the present invention s it uses a single conveyor belt of drying surface.

- The use of vacuum pumps versus condensing coils.

The invention described by US Patent No. 4,033,048 uses a vacuum pump to extract water vapor in the drying chamber. If water condenses within the vacuum pump itself (a very likely occurrence) the pump will sustain significant damage Hence, it is probable that pumps will need to be replaced regularly if the invention were to be operated on a continual basis. The cost for an industrial size vacuum pump is approximately NZ$10,000 To replace this type of pump on a regular basis would substantially increase the running and maintenance costs of this invention.

The present invention uses condensing coils to remove the water from the spray frozen material. A vacuum pump is only employed to remove incondensable material and to counterbalance vacuum leaks rather than to remove water from the spray frozen product.

- The method of heating.

  The invention disclosed in US Patent No. 4,033,048 requires considerable microwave insulation and the use of a microwave "choke" This makes the invention disclosed in this patent considerably more complex to manufacture, maintain and run.

  The present application does not require the use of either of these aspects and hence uses a heating method which is considerably simpler than that disclosed in this United States patent

Further this patent does not teach a method of spray-freeze drying which is characterised by controlling the thickness of frozen product. It does clearly not anticipate the present invention.

CONCLUSION

The present application is for a method of spray freeze drying which is characterised by controlling the thickness of product formed by spray freezing This acts to improve the efficiency and quality of the product produced by the invention It also removes any problems associated with the damage of product by heat during the drying process.

The cited documents, either alone or in combination, do not disclose the present invention. The present invention is clearly both novel and inventive.

We trust that with these amendments and submissions the examiner will now agree that this invention is both novel and inventive. We look forward to receiving a favourable written opinion in due course.

Yours sincerely
JAMES & WELLS

pp

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THE CLAIMS DEFINING THE INVENTION ARE:

1 A method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein including the steps of:

holding a chamber at a temperature and pressure below the triple-point of the liquid substance,

injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

collecting the FLS portion as a layer on a surface, and

conveying the collected FLS portion on the surface,

wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface.

2 A method as claimed in claim 1, wherein the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface

3. A method as claimed in claim 1 or claim 2, wherein the FEL is condensed by a condensing device

4. A method as claimed in anyone of claims 1 to 3, wherein the FEL is removed from said chamber

5 A method as claimed in any one of the preceding claims, wherein injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS.

6. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising of the liquid substance is performed by one or more nozzles.

7. A method as claimed in any one of the preceding claims, wherein the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface.

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8 A method as claimed in claim 7, wherein the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion

9 A method as claimed in claim 8, wherein the predetermined particle size is substantially 500 μm or more

10. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 500 μm.

11. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 200 μm

12 A method as claimed in any one of the preceding claims, wherein the surface conveys the FLS portion away from the one or more nozzles.

13 A method as claimed in any one of the preceding claims, wherein the method includes the step of exposing the collected layer of FLS portion to a heating means thereby substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product.

14 A method as claimed in claim 13, wherein the SEL portion is condensed by a condensing device.

15. A method as claimed in claim 13 or claim 14, wherein the SEL portion is removed from the chamber.

16 A method as claimed in any one of claims 13 to 15, wherein the product is removed from the conveying surface by a product removal device.

17. A method as claimed in any one of claims 13 to 16, wherein the product is removed from the chamber via the outlet port.

18. A method as claimed in any one of the preceding claims, wherein the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein.

19 A method as claimed in any one of the preceding claims where the substance is selected from one or more of the following: a slurry of coffee, liquid milk, fruit and/or vegetable juices

20. An apparatus for drying or concentrating a liquid substance comprising

a chamber and a chamber pressure reduction device, and

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one or more injection ports through which the liquid substance is injected to the chamber,

a collection surface which collects a frozen liquid substance portion of the liquid substance,

wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled.

21 An apparatus as claimed in claim 20, wherein the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface

22 An apparatus as claimed in claim 20 or claim 21, wherein the FEL portion is condensed by a condensing device.

23. An apparatus as claimed in any one of claims 20 to 22, wherein said FEL is removed from said chamber.

24 An apparatus as claimed in any one claims 20 to claim 23, wherein the one or more injection ports comprises at least one spray or atomisation nozzle.

25. An apparatus as claimed in claim 24, wherein the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle size

26 An apparatus as claimed in any one of claims 20 to 25, wherein the pressure reduction device is a gas evacuation pump.

27. An apparatus as claimed in any one of claims 20 to 26, wherein the condensing device is a cooled coil or coils

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28. An apparatus as claimed in any one of claims 20 to 27, wherein the condensing device is cooled with a refrigerant.

29 An apparatus as claimed in any one of claims 20 to 28, wherein the collection surface includes one, or a combination of, the following conveyors: a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray

30 An apparatus as claimed in claim 29, wherein the angled tray and/or vibrating tray includes a reduced friction surface.

31. An apparatus as claimed in claim 30, wherein the reduced friction surface comprises polytetrafluroethylene (PTFE).

32 An apparatus as claimed in any one of claims 20 to 31, wherein the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports.

33 An apparatus as claimed in claim 32, wherein the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product

34 An apparatus as claimed in claim 32 or claim 33, wherein the heating means is one or a combination of the following energy sources: infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface.

35 An apparatus as claimed in claim 33 or claim 34, wherein a product removal device is employed to remove the product from the collection surface.

36 An apparatus as claimed in claim 35, wherein the product removal device is a scraping means and/or a brushing means.

37. An apparatus as claimed in claim 36, wherein the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port

38 An apparatus as claimed in claim 36, wherein the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port

39 An apparatus as claimed in any one of claims 33 to 37, wherein the product is removed via a chamber outlet port.

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40 An apparatus as claimed in any one of claims 20 to 39, wherein the chamber is held at a pressure of substantially 611.3 Pa or less

41. A drying process for a liquid substance comprising the steps of:

atomising the liquid substance,

cooling the atomised liquid substance (ALS) to initiate a phase change,

conveying the ALS into a drying chamber under a vacuum,

heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber

42 A drying process as claimed in claim 41, wherein the liquid substance is chilled from an initial temperature prior to atomising.

43 A drying process as claimed in claim 41 or claim 42, wherein the ALS conveyed into the drying chamber is also conveyed through the drying chamber

44 A drying process as claimed in any one of claims 41 to 43, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation

45 A drying process as claimed in any one of claims 42 to 44, wherein the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature.

46 A drying process as claimed in any one of claims 41 to 45, wherein atomising the liquid substance achieves a predetermined particle size.

47 A drying process as claimed in claim 46, wherein the predetermined particle size is substantially 500 μm or more

48. A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 500 μm.

49. A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 200 μm.

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50. A drying process as claimed in any one of claims 41 to 49, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

51 A drying process as claimed in any one of claims 42 to 50, wherein chilling is undertaken with direct or indirect contact with a cold fluid.

52 A drying process as claimed in claim 51, wherein said cold fluid is air.

53 A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is substantially 0°C or less.

54 A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is less than substantially -20°C.

55 A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a counter current configuration

56. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a co-current configuration.

57 A drying process as claimed in any one of claims 41 to 56, wherein the ALS is reduced to a temperature below its eutectic temperature.

58. A drying process as claimed in any one of claims 41 to 57, wherein the ALS is conveyed to a separator

59 A drying process as claimed in any one of claims 41 to 57, wherein said ALS is conveyed pneumatically to a separator

60. A drying process as claimed in claim 58 or claim 59, wherein said separator is a gas-solid separation device

61 A drying process as claimed in claim 60, wherein said gas-solid separation device is a cyclone

62. A drying process as claimed in claim 60 or claim 61, wherein the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step

63. A drying process as claimed in claim 61 or claim 62, wherein one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet.

64. A drying process as claimed in any one of claims 60 to 63, wherein separated solids from said gas-solid separation device enter a vacuumous drying chamber

65 A drying process as claimed in any one of claims 41 to 64, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device.

66 A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less

67 A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

68 A drying process as claimed in any one of claims 41 to 67, wherein said ALS is conveyed via a surface

69 A drying process as claimed in any one of claims 41 to 68, wherein said ALS is conveyed via a vibrating surface

70 A drying process as claimed in claim 69, wherein said vibrating surface is a vibrating tray

71 A drying process as claimed in claim 69 or claim 70, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

72 A drying process as claimed in claim 70 or claim 71, wherein said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation

73. A drying process as claimed in any one of claims 41 to 72, wherein a vapour produced by sublimation is removed from the drying chamber.

74 A drying process as claimed in claim 73, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers

75. A drying process as claimed in any one of claims 41 to 74, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

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76 A drying process as claimed in any one of claims 41 to 75, wherein said temperature gradient is provided by an energy source.

77 A drying process as claimed in claim 76, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater

78 A drying process as claimed in any one of claims 41 to 77, wherein atomising the liquid substance achieves a predetermined particle size.

79 A drying process as claimed in claim 78, wherein the predetermined particle size is substantially 500 μm or more

80. A drying process as claimed in claim 78, wherein the predetermined particle size is less than substantially 500 μm.

81 A drying process as claim in claim 78, wherein the predetermined particle size is less than substantially 200 μm.

82 A drying process as claimed in any one of claims 60 to 81, wherein a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber.

83. A drying process as claimed in any one of claims 41 to 82, wherein a product produced is substantially reduced is free of liquid compared to the liquid substance.

84. An apparatus for a drying process for a liquid substance comprising:

an atomiser capable of atomising a liquid substance,

a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,

a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,

an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and

a collector capable of collecting the dried ALS

85 An apparatus for a drying a drying process as claimed in claim 84, wherein a

chiller capable of chilling a liquid substance from an initial temperature to a lower temperature is provided prior to the liquid substance atomiser.

86. An apparatus for a drying a drying process as claimed in claim 84 or claim 85, wherein the apparatus includes a conveyor capable of conveying the ALS through the drying chamber

87 An apparatus for a drying a drying process as claimed in any one of claims 84 to 86, wherein the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation.

88. An apparatus for a drying process as claimed in claim 87, wherein the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber.

89. An apparatus for a drying process as claimed in any one of claims 84 to 88, wherein atomising the liquid substance achieves a predetermined particle size.

90 An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is substantially 500 μm or more.

91 An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is less than substantially 500 μm.

92 An apparatus for a drying process as claimed in claim 89, wherein the predetermined size is less than substantially 200 μm

93. An apparatus for a drying process as claimed in any one of claims 84 to 92, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS)

94. An apparatus for a drying process as claimed in claim 93, wherein said cold gas is air

95. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is substantially 0°C or less.

96. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is less than substantially -20°C.

97 An apparatus for a drying process as claimed in any one of claims 84 to 96, wherein the vacuum of said vacuumous drying chamber is created by a

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pressure reduction device.

98 An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less

99 An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure

100 An apparatus for a drying process as claimed in any one of claims 84 to 99, wherein said ALS is conveyed via a vibrating surface

101 An apparatus for a drying process as claimed in claim 100, wherein said vibrating surface is a vibrating tray

102 An apparatus for a drying process as claimed in claim 101, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

103 An apparatus for a drying process as claimed in claim 84 or claim 102, wherein ALS is conveyed through a temperature gradient to substantially effect sublimation.

104 An apparatus for a drying process as claimed in claim 84 or claim 103, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers

105 An apparatus for a drying process as claimed in claim 103, wherein said temperature gradient is provided by an energy source.

106. An apparatus for a drying process as claimed in claim 105, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

107 A drying chamber for use with the apparatus as claimed in any one of claims 20 to 40 which comprises, Deleted: ,

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

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a heat source adapted to act upon said tray.

108. A drying chamber for use with the apparatus as claimed in anyone of claims 84 to 106 which comprises.

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray

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ABSTRACT

This invention relates to a process and/or method and associated apparatus for use in spray freeze drying a fluid substance such as fruit juice, pharmaceuticals, nutraceuticals, tea and coffee. A fluid substance is sprayed (atomised) and reduced in temperature to below its eutectic temperature; thereby inducing a phase change of fluids in the fluid substance. The atomised fluid substance (AFS) is then subjected to a vacuumous drying chamber in which the AFS is subjected to an energy source to assist sublimation of fluid from the AFS as it is conveyed through the chamber. Such an energy source can provide a temperature gradient through which the AFS passes and is exposed to, all carried out so as to preferably minimise or reduce the likelihood of degradation of the substance being dried (reducing the fluid content).

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THE CLAIMS DEFINING THE INVENTION ARE:

1. A method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein including the steps of

   holding a chamber at a temperature and pressure below the triple-point of the liquid substance,

   injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

   collecting the FLS portion as a layer on a surface, and

   conveying the collected FLS portion on the surface,

   wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface

2. A method as claimed in claim 1, wherein the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

3 A method as claimed in claim 1 or claim 2, wherein the FEL is condensed by a condensing device.

4. A method as claimed in anyone of claims 1 to 3, wherein the FEL is removed from said chamber.

5. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS.

6. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising of the liquid substance is performed by one or more nozzles.

7. A method as claimed in any one of the preceding claims, wherein the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface.

8. A method as claimed in claim 7, wherein the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion.

9. A method as claimed in claim 8, wherein the predetermined particle size is substantially 500 μm or more.

10. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 500 μm

11. A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 200 μm.

12. A method as claimed in any one of the preceding claims, wherein the surface conveys the FLS portion away from the one or more nozzles.

13. A method as claimed in any one of the preceding claims, wherein the method includes the step of exposing the collected layer of FLS portion to a heating means thereby substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product.

14. A method as claimed in claim 13, wherein the SEL portion is condensed by a condensing device.

15. A method as claimed in claim 13 or claim 14, wherein the SEL portion is removed from the chamber.

16. A method as claimed in any one of claims 13 to 15, wherein the product is removed from the conveying surface by a product removal device.

17 A method as claimed in any one of claims 13 to 16, wherein the product is removed from the chamber via the outlet port.

18. A method as claimed in any one of the preceding claims, wherein the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein

19. A method as claimed in any one of the preceding claims where the substance is selected from one or more of the following: a slurry of coffee, liquid milk, fruit and/or vegetable juices.

20. An apparatus for drying or concentrating a liquid substance comprising:

a chamber and a chamber pressure reduction device, and

one or more injection ports through which the liquid substance is injected to the chamber,

a collection surface which collects a frozen liquid substance portion of the liquid substance,

wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled.

21. An apparatus as claimed in claim 20, wherein the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface

22. An apparatus as claimed in claim 20 or claim 21, wherein the FEL portion is condensed by a condensing device.

23. An apparatus as claimed in any one of claims 20 to 22, wherein said FEL is removed from said chamber.

24. An apparatus as claimed in any one claims 20 to claim 23, wherein the one or more injection ports comprises at least one spray or atomisation nozzle.

25 An apparatus as claimed in claim 24, wherein the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle size.

26. An apparatus as claimed in any one of claims 20 to 25, wherein the pressure reduction device is a gas evacuation pump

27. An apparatus as claimed in any one of claims 20 to 26, wherein the condensing device is a cooled coil or coils.

28. An apparatus as claimed in any one of claims 20 to 27, wherein the condensing device is cooled with a refrigerant.

29. An apparatus as claimed in any one of claims 20 to 28, wherein the collection surface includes one, or a combination of, the following conveyors: a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray.

30 An apparatus as claimed in claim 29, wherein the angled tray and/or vibrating tray includes a reduced friction surface.

31 An apparatus as claimed in claim 30, wherein the reduced friction surface comprises polytetrafluroethylene (PTFE).

32. An apparatus as claimed in any one of claims 20 to 31, wherein the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports.

33. An apparatus as claimed in claim 32, wherein the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product.

34. An apparatus as claimed in claim 32 or claim 33, wherein the heating means is one or a combination of the following energy sources: infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface.

35. An apparatus as claimed in claim 33 or claim 34, wherein a product removal device is employed to remove the product from the collection surface.

36. An apparatus as claimed in claim 35, wherein the product removal device is a scraping means and/or a brushing means.

37. An apparatus as claimed in claim 36, wherein the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port.

38. An apparatus as claimed in claim 36, wherein the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port.

39. An apparatus as claimed in any one of claims 33 to 37, wherein the product is removed via a chamber outlet port.

40. An apparatus as claimed in any one of claims 20 to 39, wherein the chamber is held at a pressure of substantially 611 3 Pa or less

41. A drying process for a liquid substance comprising the steps of:

atomising the liquid substance,

cooling the atomised liquid substance (ALS) to initiate a phase change,

conveying the ALS into a drying chamber under a vacuum,

heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber.

42. A drying process as claimed in claim 41, wherein the liquid substance is chilled from an initial temperature prior to atomising.

43. A drying process as claimed in claim 41 or claim 42, wherein the ALS conveyed into the drying chamber is also conveyed through the drying chamber

44. A drying process as claimed in any one of claims 41 to 43, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

45. A drying process as claimed in any one of claims 42 to 44, wherein the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature.

46 A drying process as claimed in any one of claims 41 to 45, wherein atomising the liquid substance achieves a predetermined particle size.

47. A drying process as claimed in claim 46, wherein the predetermined particle size is substantially 500 μm or more.

48 A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 500 μm.

49 A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 200 μm.

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50 A drying process as claimed in any one of claims 41 to 49, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

51. A drying process as claimed in any one of claims 42 to 50, wherein chilling is undertaken with direct or indirect contact with a cold fluid

52 A drying process as claimed in claim 51, wherein said cold fluid is air

53 A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is substantially 0°C or less.

54. A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is less than substantially -20°C.

55. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a counter current configuration

56. A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a co-current configuration.

57. A drying process as claimed in any one of claims 41 to 56, wherein the ALS is reduced to a temperature below its eutectic temperature.

58. A drying process as claimed in any one of claims 41 to 57, wherein the ALS is conveyed to a separator.

59 A drying process as claimed in any one of claims 41 to 57, wherein said ALS is conveyed pneumatically to a separator

60 A drying process as claimed in claim 58 or claim 59, wherein said separator is a gas-solid separation device.

61. A drying process as claimed in claim 60, wherein said gas-solid separation device is a cyclone.

62. A drying process as claimed in claim 60 or claim 61, wherein the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step.

63. A drying process as claimed in claim 61 or claim 62, wherein one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet

64. A drying process as claimed in any one of claims 60 to 63, wherein separated solids from said gas-solid separation device enter a vacuumous drying chamber

65 A drying process as claimed in any one of claims 41 to 64, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device

66 A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less

67. A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

68 A drying process as claimed in any one of claims 41 to 67, wherein said ALS is conveyed via a surface.

69. A drying process as claimed in any one of claims 41 to 68, wherein said ALS is conveyed via a vibrating surface.

70. A drying process as claimed in claim 69, wherein said vibrating surface is a vibrating tray

71 A drying process as claimed in claim 69 or claim 70, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

72 A drying process as claimed in claim 70 or claim 71, wherein said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation.

73. A drying process as claimed in any one of claims 41 to 72, wherein a vapour produced by sublimation is removed from the drying chamber

74. A drying process as claimed in claim 73, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers.

75. A drying process as claimed in any one of claims 41 to 74, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

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76 A drying process as claimed in any one of claims 41 to 75, wherein said temperature gradient is provided by an energy source.

77 A drying process as claimed in claim 76, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

78 A drying process as claimed in any one of claims 41 to 77, wherein atomising the liquid substance achieves a predetermined particle size.

79. A drying process as claimed in claim 78, wherein the predetermined particle size is substantially 500 μm or more.

80 A drying process as claimed in claim 78, wherein the predetermined particle size is less than substantially 500 μm

81 A drying process as claim in claim 78, wherein the predetermined particle size is less than substantially 200 μm.

82. A drying process as claimed in any one of claims 60 to 81, wherein a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber.

83. A drying process as claimed in any one of claims 41 to 82, wherein a product produced is substantially reduced is free of liquid compared to the liquid substance.

84. An apparatus for a drying process for a liquid substance comprising:

an atomiser capable of atomising a liquid substance,

a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,

a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,

an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and

a collector capable of collecting the dried ALS.

85. An apparatus for a drying a drying process as claimed in claim 84, wherein a

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chiller capable of chilling a liquid substance from an initial temperature to a lower temperature is provided prior to the liquid substance atomiser.

86. An apparatus for a drying a drying process as claimed in claim 84 or claim 85, wherein the apparatus includes a conveyor capable of conveying the ALS through the drying chamber.

87. An apparatus for a drying a drying process as claimed in any one of claims 84 to 86, wherein the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation.

88. An apparatus for a drying process as claimed in claim 87, wherein the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber

89. An apparatus for a drying process as claimed in any one of claims 84 to 88, wherein atomising the liquid substance achieves a predetermined particle size.

90 An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is substantially 500 μm or more

91 An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is less than substantially 500 μm.

92. An apparatus for a drying process as claimed in claim 89, wherein the predetermined size is less than substantially 200 μm.

93. An apparatus for a drying process as claimed in any one of claims 84 to 92, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

94. An apparatus for a drying process as claimed in claim 93, wherein said cold gas is air

95. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is substantially 0°C or less.

96. An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is less than substantially -20°C.

97. An apparatus for a drying process as claimed in any one of claims 84 to 96, wherein the vacuum of said vacuumous drying chamber is created by a

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pressure reduction device.

98. An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

99 An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

100. An apparatus for a drying process as claimed in any one of claims 84 to 99, wherein said ALS is conveyed via a vibrating surface

101. An apparatus for a drying process as claimed in claim 100, wherein said vibrating surface is a vibrating tray.

102. An apparatus for a drying process as claimed in claim 101, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven.

103 An apparatus for a drying process as claimed in claim 84 or claim 102, wherein ALS is conveyed through a temperature gradient to substantially effect sublimation

104 An apparatus for a drying process as claimed in claim 84 or claim 103, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers.

105. An apparatus for a drying process as claimed in claim 103, wherein said temperature gradient is provided by an energy source.

106. An apparatus for a drying process as claimed in claim 105, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater.

107 A drying chamber for use with the apparatus as claimed in any one of claims 20 to 40 which comprises:

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

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a heat source adapted to act upon said tray

108. A drying chamber for use with the apparatus as claimed in anyone of claims 84 to 106 which comprises:

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray.

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ABSTRACT

This invention relates to a process and/or method and associated apparatus for use in spray freeze drying a fluid substance such as fruit juice, pharmaceuticals, nutraceuticals, tea and coffee. A fluid substance is sprayed (atomised) and reduced in temperature to below its eutectic temperature; thereby inducing a phase change of fluids in the fluid substance The atomised fluid substance (AFS) is then subjected to a vacuumous drying chamber in which the AFS is subjected to an energy source to assist sublimation of fluid from the AFS as it is conveyed through the chamber. Such an energy source can provide a temperature gradient through which the AFS passes and is exposed to, all carried out so as to preferably minimise or reduce the likelihood of degradation of the substance being dried (reducing the fluid content)

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PATENT COOPERATION TREATY

From the:
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To. James & Wells Private Bag 3140 Hamilton NEW ZEALAND	**PCT** WRITTEN OPINION OF THE INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY (PCT Rule 66)
	Date of mailing (day/month/year) 26 APR 2006
Applicant's or agent's file reference 230677X374	REPLY DUE within TWO MONTHS from the above date of mailing

International application No.	International filing date (day/month/year)	Priority date (day/month/year)
PCT/NZ2005/000089	2 May 2005	1 May 2004

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

INT. CL.

B01D 1/18 (2006.01) ***F26B 5/06*** (2006 01)

ACTION DATE: 18 APRIL 2006

Applicant

AGRESEARCH LIMITED et al

1. [X] The written opinion established by the International Searching Authority
   [X] is [ ] is not
   considered to be a written opinion of the International Preliminary Examining Authority.
2. This **second** (second, etc.) opinion contains indications relating to the following items
   - [X] Box No. I Basis of the opinion
   - [ ] Box No II Priority
   - [ ] Box No III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - [ ] Box No IV Lack of unity of invention
   - [X] Box No. V Reasoned statement under Rule 66 2(a)(ii) with regard to novelty, inventive step or industrial applicability, citations and explanations supporting such statement
   - [ ] Box No VI Certain documents cited
   - [ ] Box No. VII Certain defects in the international application
   - [ ] Box No VIII Certain observations on the international application
3. The applicant is hereby **invited to reply** to this opinion.

   When? See the Reply Due date indicated above. However, the Australian Patent Office will not establish the Report before the earlier of (i) a response being filed, or (ii) one month before the Final Date by which the international preliminary examination report must be established The Report will take into account any response (including amendments) filed before the Report is established.
   If no response is filed by 1 month before the Final Date, the international preliminary examination report will be established on the basis of this opinion
   Applicants wishing to have the benefit of a further opinion (if needed) before the report is established should ensure that a response is filed at least 3 months before the Final Date by which the international preliminary examination report must be established.

   How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3.
   For the form and the language of the amendments, see Rules 66.8 and 66 9.

   Also For an additional opportunity to submit amendments, see Rule 66.4.
   For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4*bis*
   For an informal communication with the examiner, see Rule 66.6.
4. The **FINAL DATE** by which the international preliminary report on patentability (Chapter II of the PCT) must be established according to Rule 69 2 is: 1 September 2006

Name and mailing address of the IPEA/AU	Authorized Officer
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address. pct@ipaustralia gov au Facsimile No (02) 6285 3929	**THARU FERNANDO** Telephone No. (02) 6283 2486

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WRITTEN OPINION OF THE INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY	International application No. PCT/NZ2005/000089

Box No. I **Basis of the opinion**

1. With regard to the language, this opinion has been established on the basis of:

[X] The international application in the language in which it was filed:

[ ] A translation of the international application into , which is the language of a translation furnished for the purposes of :

[ ] international search (under Rules 12.3(a) and 23.1 (b))

[ ] publication of the international application (under Rule 12.4(a))

[ ] international preliminary examination (Rules 55.2(a) and/or 55.3(a))

2. With regard to the elements of the international application, this opinion has been established on the basis of *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed")*:

[ ] the international application as originally filed/furnished

[X] the description: pages 1-21, as originally filed/furnished
pages , received by this Authority on with the letter of
pages , received by this Authority on with the letter of

[X] the claims: pages , as originally filed/furnished
pages , as amended (together with any statement) under Article 19,
pages 22-32, received by this Authority on 23 December 2005 with the letter of 23 December 2005
pages , received by this Authority on with the letter of

[X] the drawings: pages 1-4, as originally filed/furnished
pages , received by this Authority on with the letter of
pages , received by this Authority on with the letter of

[ ] a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing.

3. [ ] The amendments have resulted in the cancellation of:

[ ] the description, pages
[ ] the claims, Nos
[ ] the drawings, sheets/figs
[ ] the sequence listing *(specify)*:
[ ] any table(s) related to the sequence listing *(specify)*:

4 [ ] This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

[ ] the description, pages
[ ] the claims, Nos.
[ ] the drawings, sheets/figs
[ ] the sequence listing *(specify)*.
[ ] any table(s) related to the sequence listing *(specify)*:

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WRITTEN OPINION OF THE
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

International application No.
PCT/NZ2005/000089

Box No. V **Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims	**20-40**	YES
	Claims	**1-19, 41-108**	NO
Inventive step (IS)	Claims	**20-40**	YES
	Claims	**1-19, 41-108**	NO
Industrial applicability (IA)	Claims	**1-108**	YES
	Claims		NO

2. Citations and explanations:

D3: US 4033048

<u>Novelty (N) and Inventive Step (IS)</u> (Claims 1-108)

Claim 1 is directed to a method of drying/concentrating a liquid solution by injecting and/or atomising the liquid solution into a chamber that is at a temperature and pressure below the triple point of the liquid solution. This generates a frozen liquid substance portion (FLS) and a first evaporated liquid substance portion (FEL). The FLS portion is collected as a layer on a surface and is conveyed at a rate that controls the thickness of the layer of collected FLS on the surface.

This is anticipated by D3, which discloses spraying a liquid product (*atomised liquid*) into a freezing chamber allowing a powder to form (*FLS*) which then falls onto a conveyor surface in a drying chamber. Heat is also applied allowing excess moisture to sublimate. Further, the entire interior of the apparatus is under vacuum.

Further, claims 2-19 are also anticipated by the document (see especially col 3-4).

Claim 41 is directed to a drying process for a liquid substance wherein the liquid substance is atomised, cooled to initiate a phase change, conveyed into a drying chamber under vacuum, heated so as to effect sublimation and then collecting the substantially dried atomised liquid substance from the chamber.

Similarly, D3, also anticipates this claim, disclosing the spraying a liquid product (*atomised liquid*) into a freezing chamber allowing a powder to form (*phase change*) which then falls onto a conveyor surface in a drying chamber. Heat is also applied allowing excess moisture to sublimate (*heated so as to effect sublimation*) and then the dried product us collected in a collection chamber.

Appended claims 42-108 are also anticipated by this document.

Please note that the attorney's response has been considered, and although there may be numerous differences between the <u>description</u> of the present invention and D3, the <u>claims</u> of the present invention, as mentioned above, are drafted so as to encompass the invention of D3.

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To: +61 2 6285 3929

Total Pages: 1

26 May 2006
HAMILTON OFFICE

IP Australia
PO Box 200
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Attention: Neeti Saxena

Re: AGRESEARCH LIMITED
PCT Application No. PCT/NZ2005/000089
DRYING PROCESS AND APPARATUS
Our Ref: 230677/80 RTM

We refer to the examiner's written opinion dated 26 April 2006 and matters arising.

The present invention is a method of drying or concentrating a liquid substance. It is true that this is in the same area of invention as that disclosed in US Patent No. 4,03,3048. However, the present invention is characterised by controlling the thickness of spray frozen liquid substances formed on a drying surface. This is clearly a novel aspect of the present invention which is reflected in claim 1 of the specification.

We note that the examiner believes US Patent No. 4033048 anticipates this aspect of the present invention. However, nowhere in this document is there mention of controlling the thickness of the spray frozen material.

It is true that this patent does mention spray frozen material falling as a "snow like powder". However, this is into a collection funnel where it is then transferred to a further drying surface. The cited document does not teach a method to control the rate of spray frozen material on a drying surface and it does not disclose the present invention.

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James & Wells Intellectual Property comprises the partnerships of:
[1] James & Wells Lawyers; and
[2] James & Wells Patent & Trade Mark Attorneys

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INTELLECTUAL PROPERTY

Accordingly, the claims of the present application clearly define a novel invention. Namely, a method characterised by controlling the thickness of spray frozen substance formed on a drying surface.

If this aspect were not included in the invention it is unlikely that it would be successful. It is essential to allow the invention to function efficiently and successfully.

The examiner has accepted that there are numerous differences between the description of the present invention and United States Patent No. 4,033,048. However, the claims of the present application do not overlap with those of the cited documents and do reflect the differences between the inventions.

Further, the examiner has accepted that claims 20 to 40 are novel and inventive. These claims contain a similar provision to that in claim 1. Namely, that the present invention is inventive because it allows the thickness of frozen product to be controlled.

With regard to the foregoing, we trust the examiner will now agree that the invention is both novel and inventive, and look forward to receiving a favorable Written Opinion in due course.

Yours sincerely
JAMES & WELLS

David Macaskill / Kate Wilson
IP Specialist / Partner
davidmac@jaws.co.nz / katew@jaws.co.nz

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PATENT COOPERATION TREATY

From the:
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:	**PCT**
James & Wells Private Bag 3140 Hamilton NEW ZEALAND	WRITTEN OPINION OF THE INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY (PCT Rule 66)
	Date of mailing *(day/month/year)* 20 JUN 2006
Applicant's or agent's file reference 230677X374	**REPLY DUE** within **TWO MONTHS** from the above date of mailing

International application No	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/NZ2005/000089	2 May 2005	1 May 2004

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

INT. CL.

B01D 1/18 (2006.01) ***F26B 5/06*** (2006.01)

ACTION DATE: 14 JUNE 2006

Applicant

AGRESEARCH LIMITED et al

1. [X] The written opinion established by the International Searching Authority:

   [X] is [ ] is not

   considered to be a written opinion of the International Preliminary Examining Authority.

2. This **third** (second, etc.) opinion contains indications relating to the following items:

   - [X] Box No I Basis of the opinion
   - [ ] Box No II Priority
   - [ ] Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - [ ] Box No. IV Lack of unity of invention
   - [X] Box No V Reasoned statement under Rule 66 2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - [ ] Box No. VI Certain documents cited
   - [ ] Box No VII Certain defects in the international application
   - [ ] Box No VIII Certain observations on the international application

3. The applicant is hereby invited to reply to this opinion.

   When? See the **Reply Due** date indicated above. However, the Australian Patent Office will not establish the Report before the earlier of (i) a response being filed, or (ii) one month before the **Final Date** by which the international preliminary examination report must be established The Report will take into account any response (including amendments) filed before the Report is established.
   If no response is filed by 1 month before the Final Date, the international preliminary examination report will be established on the basis of this opinion
   Applicants wishing to have the benefit of a further opinion (if needed) before the report is established should ensure that a response is filed at least **3 months before the Final Date** by which the international preliminary examination report must be established

   How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66 3.
   For the form and the language of the amendments, see Rules 66 8 and 66 9.

   Also For an additional opportunity to submit amendments, see Rule 66 4.
   For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4*bis*
   For an informal communication with the examiner, see Rule 66.6.

4. The **FINAL DATE** by which the international preliminary report on patentability (Chapter II of the PCT) must be established according to Rule 69.2 is. 1 September 2006

Name and mailing address of the IPEA/AU	Authorized Officer
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia gov au Facsimile No. (02) 6285 3929	**THARU FERNANDO** Telephone No. (02) 6283 2486

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WRITTEN OPINION OF THE
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

International application No.
PCT/NZ2005/000089

Box No. I **Basis of the opinion**

1. With regard to the **language**, this opinion has been established on the basis of:

[X] The international application in the language in which it was filed:

[ ] A translation of the international application into , which is the language of a translation furnished for the purposes of:

[ ] international search (under Rules 12 3(a) and 23.1 (b))

[ ] publication of the international application (under Rule 12 4(a))

[ ] international preliminary examination (Rules 55.2(a) and/or 55 3(a))

2. With regard to the elements of the international application, this opinion has been established on the basis of *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed.")*.

[ ] the international application as originally filed/furnished

[X] the description pages **1-21**, as originally filed/furnished
pages , received by this Authority on with the letter of
pages , received by this Authority on with the letter of

[X] the claims. pages , as originally filed/furnished
pages , as amended (together with any statement) under Article 19,
pages **22-32**, received by this Authority on **23 December 2005** with the letter of **23 December 2005**
pages , received by this Authority on with the letter of

[X] the drawings: pages **1-4**, as originally filed/furnished
pages , received by this Authority on with the letter of
pages , received by this Authority on with the letter of

[ ] a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing

3. [ ] The amendments have resulted in the cancellation of:

[ ] the description, pages
[ ] the claims, Nos
[ ] the drawings, sheets/figs
[ ] the sequence listing *(specify)*:
[ ] any table(s) related to the sequence listing *(specify)*:

4. [ ] This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

[ ] the description, pages
[ ] the claims, Nos.
[ ] the drawings, sheets/figs
[ ] the sequence listing *(specify)*:
[ ] any table(s) related to the sequence listing *(specify)*:

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WRITTEN OPINION OF THE
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

International application No.
PCT/NZ2005/000089

Box No. V **Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims **1-40**	**YES**
	Claims **41-108**	**NO**
Inventive step (IS)	Claims **1-40**	**YES**
	Claims **41-108**	**NO**
Industrial applicability (IA)	Claims **1-108**	**YES**
	Claims	**NO**

2. Citations and explanations:

D3: US 4033048

Novelty (N) and Inventive Step (IS) (Claims 1-108)

Claim 41 is directed to a drying process for a liquid substance wherein the liquid substance is atomised, cooled to initiate a phase change, conveyed into a drying chamber under vacuum, heated so as to effect sublimation and then collecting the substantially dried atomised liquid substance from the chamber.

D3 anticipates this claim by disclosing spraying a liquid product (*atomised liquid*) into a freezing chamber, allowing a powder to form (*phase change*) which then falls onto a conveyor surface in a drying chamber. Heat is also applied allowing excess moisture to sublimate (*heated so as to effect sublimation*) and then the dried product is collected in a collection chamber.

Appended claims 42-108 are also anticipated by this document.

According to the attorney's response, the present invention is characterised by controlling the thickness of spray frozen liquid substances. The attorney further indicates that the invention is unlikely to be successful if this aspect were not included, and that this characteristic is essential in allowing the invention to function efficiently and successfully. However, claim 41 is absent on this aspect of the invention and as such is anticipated by **D3**.

Claims 1-40, which are characterised by the particular characteristic mentioned above, are novel and inventive.

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DRYING PROCESS AND APPARATUS

TECHNICAL FIELD

This invention relates to a dryer and a drying process In particular, though not solely, this invention relates to a dryer for use in freezing, concentrating or drying a liquid substance having solid particles in suspension or a substance dissolved therein

BACKGROUND ART

The process of food preservation has always been of interest, with freeze drying being of particular food industry focus since the late 1940's In the formative years of freeze drying apparatus and processing development, there was an emphasis in achieving a useful (preserved) end product The basic stages of freezing a liquid material and then heating the frozen material under vacuum to effect moisture removal are preferable to earlier attempts at food preservation which often involved dehydration by hot air convection methods

Dried product quality is of prime importance in terms of nutritional value and appearance to the consumer It is undesirable to damage the product during the processing and removal of liquids from substances. Hot air convection drying often results in product shrinkage and other detrimental effects

Batch unit operations have been used in the freeze drying industry although batch processing has a number of disadvantages which include, longer product processing durations, longer residence times at each stage of processing (often as manual labour is required to transfer partially processed substances from one stage to the next), poorly optimised processing equipment often with excess capacity, increased set-up times, reduced control of the process due to increased likelihood of human error (due to lack of automation), and low throughput

Batch systems are typically used for small production runs or where a need for process flexibility is required.

In conventional (batch freeze) drying processes at least one liquid feed is poured into shallow tray(s) (product thickness typically varies between 10 and 20mm), which are then placed on shelves in the freeze dryer The door to the batch freezer is closed and the product is frozen After the product has frozen, the trays are heated and the ice is slowly sublimed The sublimed vapour is condensed on refrigerated coils Once it is assumed that the product is dry, the product is removed manually The product exits a conventional freeze-dryer as a brittle cake, and usually requires a separate granulation stage before it can be further processed

This method of drying a moisture laden substance by freezing it then subliming off as much excess moisture as possible to produce a dried product is primarily used within industries where substances need to be dried, but are unable to withstand even moderate temperatures, for example some foodstuffs or pharmaceuticals can be damaged or affected by heat

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It is not unusual for this combined freezing and drying process to take 48 hours or more This is undesirable if heat-sensitive materials are being dried Also, if the vacuum is lost in a conventional batch freeze-dryer, melting and glass formation may occur, and it is possible that the entire load of product may be lost, that is the product may not be a useable quality product or able to be sold In addition, the loading and unloading process is susceptible to product contamination due to exposure, and wastage from spillage of the shallow trays.

Therefore, a system which enables rapid freeze drying of a moisture laden substance and which produces a suitably formed end product, such as a powder, is desirable As yet no truly continuous or semi-continuous freeze drying processes have been effectively developed by the drying industry

An apparatus able to minimise potential contamination, reduce liquid feed spillage and which may increase the liquid throughput would be advantageous to the drying industry If a liquid feed is able to be freeze dried which does also not require a necessary pre-treatment liquid feed cooling stages or subsequent dried product granulation stages, then significant problems with conventional batch freeze drying may be overcome

In more recent times, the stages of freezing and drying have been further developed to include various steps such as reducing the physical size of substances to be treated, as well as graded temperature control during the freezing and drying stages However, in practice highly controlled drying has often been difficult to implement effectively as the substances being dried are often in static tray type arrangements and therefore some substance is likely to be affected or heated more so than other parts of the substance, therefore resulting in over-dried or overheated and consequently damaged product It would therefore be advantageous to utilise a drying stage with the provision of improved heat transfer conditions to substances, such a stage being preferably coupled to a freeze dry process

It is therefore an object of the present invention to provide a dryer apparatus and/or a drying process or method which goes at least some way towards addressing the foregoing problems or to at least to provide the industry with a useful choice.

All references, including any patents or patent applications cited in this specification are hereby incorporated by reference No admission is made that any reference constitutes prior art The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinence of the cited documents It will be clearly understood that, although a number of prior art publications are referred to herein, this reference does not constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country

It is acknowledged that the term 'comprise' may, under varying jurisdictions, be attributed with either an exclusive or an inclusive meaning · For the purpose of this specification, and unless otherwise noted, the term 'comprise' shall have an inclusive meaning - i e that it will be taken to mean an inclusion of not only the listed components it directly references, but also other non-specified components or elements. This rationale will also be used when the term 'comprised' or 'comprising' is used in relation to one or more steps in a method or process

Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only

DISCLOSURE OF INVENTION

Accordingly, in a first aspect, the invention broadly consists of a method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein comprising the steps

holding a chamber at a temperature and pressure below the triple-point of the liquid substance,

injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

collecting the FLS portion as a layer on a surface, and

conveying the collected FLS portion on the surface,

wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface

A "liquid substance" is used herein and refers to a substance which has liquid flow-like properties, but has constant weight/mass in a first state The weight/mass of the liquid substance can be altered by removing components from the liquid substance, for example by evaporating liquids from the substance, leaving a more concentrated liquid substance Such liquid substances may include liquids having particles or solids suspended or dissolved in a solution, as well as compounds which can exist in a liquid state, such as or similar to water or oil

Preferably, the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface

Preferably, the FEL is condensed by a condensing device

Preferably, the FEL is removed from said chamber

Preferably, injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS

Preferably, injecting and/or atomising of the liquid substance is performed by one or more nozzles

Preferably, the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface

Preferably, the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion.

Preferably, the predetermined particle size is substantially 500 μm or more

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Preferably, the predetermined particle size is less than substantially 500 μm

Preferably, the predetermined particle size is less than substantially 200 μm

Preferably, the surface conveys the FLS portion away from the one or more nozzles

Preferably, the method includes the step of exposing the collected layer of FLS portion to a heating means thereby substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product

Preferably, the SEL portion is condensed by a condensing device

Preferably, the SEL portion is removed from the chamber

Preferably, the product is removed from the conveying surface by a product removal device

Preferably, the product is removed from the chamber via the outlet port

Preferably, the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein

Preferably, the substance is selected from one or more of the following a slurry of coffee, liquid milk, fruit and/or vegetable juices

In a second aspect, the invention broadly consists of an apparatus for drying or concentrating a liquid substance comprising

a chamber and a chamber pressure reduction device, and

one or more injection ports through which the liquid substance is injected to the chamber,

a collection surface which collects a frozen liquid substance portion of the liquid substance,

wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled

Preferably, the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface

Preferably, the FEL portion is condensed by a condensing device

Preferably, said FEL is removed from said chamber

Preferably, the one or more injection ports comprises at least one spray or atomisation nozzle

Preferably, the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle size

Preferably, the pressure reduction device is a gas evacuation pump

Preferably, the condensing device is a cooled coil or coils

Preferably, the condensing device is cooled with a refrigerant

Preferably, the collection surface includes one, or a combination of, the following conveyors a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray

Preferably, the angled tray and/or vibrating tray includes a reduced friction surface

Preferably, the reduced friction surface comprises polytetrafluroethylene (PTFE)

Preferably, the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports

Preferably, the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product

Preferably, the heating means is one or a combination of the following energy sources. infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface

Preferably, a product removal device is employed to remove the product from the collection surface

Preferably, the product removal device is a scraping means and/or a brushing means.

Preferably, the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port

Preferably, the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port

Preferably, the product is removed via a chamber outlet port

Preferably, the chamber is held at a pressure of substantially 611 3 Pa or less.

In a third aspect, the invention broadly consists of a drying process for a liquid substance comprising the steps of

atomising the liquid substance,

cooling the atomised liquid substance (ALS) to initiate a phase change,

conveying the ALS into a drying chamber under a vacuum,

heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber

Preferably, the liquid substance is chilled from an initial temperature prior to atomising

Preferably, the ALS conveyed into the drying chamber is also conveyed through the drying chamber

Preferably, the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation

Preferably, the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature

Preferably, atomising the liquid substance achieves a predetermined particle size

Preferably, the predetermined particle size is substantially 500 μm or more

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Preferably, the predetermined particle size is less than substantially 500 μm

Preferably, the predetermined particle size is less than substantially 200 μm

Preferably, a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS)

Preferably, chilling is undertaken with direct or indirect contact with a cold fluid

Preferably, said cold fluid is air

Preferably, said cold fluid is substantially 0°C or less

Preferably, said cold fluid is less than substantially -20°C

Preferably, said spray freezer operates in a counter current configuration

Preferably, said spray freezer operates in a co-current configuration

Preferably, the ALS is reduced to a temperature below its eutectic temperature

Preferably, the ALS is conveyed to a separator

Preferably, said ALS is conveyed pneumatically to a separator.

Preferably, said separator is a gas-solid separation device

Preferably, said gas-solid separation device is a cyclone

Preferably, the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step

Preferably, one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet.

Preferably, separated solids from said gas-solid separation device enter a vacuumous drying chamber

Preferably, the vacuum of said vacuumous drying chamber is created by a pressure reduction device

Preferably, the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less

Preferably, the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure

Preferably, said ALS is conveyed via a surface

Preferably, said ALS is conveyed via a vibrating surface

Preferably, said vibrating surface is a vibrating tray

Preferably, said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

Preferably, said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation

Preferably, a vapour produced by sublimation is removed from the drying chamber

Preferably, vapour produced by sublimation is removed from the drying chamber by one or more condensers

Preferably, the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation

Preferably, said temperature gradient is provided by an energy source

Preferably, said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater

Preferably, atomising the liquid substance achieves a predetermined particle size

Preferably, the predetermined particle size is substantially 500 µm or more

Preferably, the predetermined particle size is less than substantially 500 µm

Preferably, the predetermined particle size is less than substantially 200 µm

Preferably, a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber

Preferably, a product produced is substantially reduced is free of liquid compared to the liquid substance

In a fourth aspect, the invention broadly consists of an apparatus for a drying process for a liquid substance comprising

an atomiser capable of atomising a liquid substance,

a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,

a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,

an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and

a collector capable of collecting the dried ALS

Preferably, a chiller capable of chilling a liquid substance from an initial temperature to a lower temperature is provided prior to the liquid substance atomiser

Preferably, the apparatus includes a conveyor capable of conveying the ALS through the drying chamber

Preferably, the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation

Preferably, the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber

Preferably, atomising the liquid substance achieves a predetermined particle size

Preferably, the predetermined particle size is substantially 500 µm or more

Preferably, the predetermined particle size is less than substantially 500 µm

Preferably, the predetermined size is less than substantially 200 µm

Preferably, a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS)

Preferably, said cold gas is air

Preferably, said cold gas is substantially 0°C or less

Preferably, said cold gas is less than substantially -20°C

Preferably, the vacuum of said vacuumous drying chamber is created by a pressure reduction device

Preferably, the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less.

Preferably, the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure

Preferably, said ALS is conveyed via a vibrating surface

Preferably, said vibrating surface is a vibrating tray

Preferably, said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

Preferably, ALS is conveyed through a temperature gradient to substantially effect sublimation

Preferably, vapour produced by sublimation is removed from the drying chamber by one or more condensers.

Preferably, said temperature gradient is provided by an energy source

Preferably, said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater

In a fifth aspect, the invention broadly consists of a drying chamber which comprises,

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray

Advantageously, the present invention provides an improved method/process of treating a liquid substance to form a dryer or at least more concentrated form of the liquid substance (in the form of a 'product)

In preferred embodiments, the present invention is able to be operated on a semi-continuous basis, or at least which will allow a greater throughput of liquid substance than compared with prior art drying systems The rate of conveyance of atomised liquid substance (ALS) is sufficient to increase efficiencies of drying/concentrating and/or which helps to reduce damage to the product

BRIEF DESCRIPTION OF DRAWINGS

Further aspects of the present invention will become apparent from the following description which is given by way of example only and with reference to the accompanying drawings in which

Figure 1 illustrates one embodiment of a possible dryer configuration according to the present invention in the third and fourth aspects

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<u>Figure 2</u>	is a process flow diagram of one possible embodiment of the present invention according to the first and second aspects,
<u>Figure 3</u>	is a top view of of the drying chamber configuration shown in Figure 2,
<u>Figure 4</u>	is a side view of the drying chamber configuration shown in Figures 2 and 3, and
<u>Figure 5</u>	is an end view of the drying chamber configuration shown in Figures 2 to 4

<u>BEST MODES FOR CARRYING OUT THE INVENTION</u>

The present invention may now be described with reference to Figures 1 to 5, and the accompanying description below

The present invention relates to process for freeze drying or dehydrating/concentrating liquid substances The process is applicable to liquids having a solid substance in suspension (for example milk) or to liquid solutions in which a substance has been dissolved Tea, coffee, fruit juice, pharmaceuticals and nutraceuticals could also be processed using this spray freeze dryer

Freeze drying is a useful preservation technique and among many other products, the following may be dried in this manner, instant coffee, vegetables for dried soup mixes, mushrooms, herbs, spices, cheese starter cultures, shrimp, fruits for ready-to-eat breakfast cereals, nutraceuticals, pharmaceuticals and agriceuticals

Some specific end users of freeze dried products may include the production of military and/or space rations as well as light weight camping foods containing vegetables, meat, fish, and fruits The freeze drying process may generally have the following advantages, low thermal damage, good retention of volatile flavours, good vitamin retention, rapid product rehydration, low product shrinkage, long product storage life – if suitably packed, retention of biological activity Although there are also some disadvantages associated with freeze drying substances may be most notably, high drying cost, damage to certain products by initial freezing, rapid deterioration unless packed and maintained at low humidity, friability (i e crumbles easily), pre-treatment sometimes necessary (e g. carrots) to avoid colour loss

A process 1 as generally outlined in the third aspect, and apparatus as broadly defined by the fourth aspect described above, comprises holding a chamber 2 at a temperature and pressure below the triple-point of a liquid substance 3, injecting the liquid substance 3 into the chamber 2 thereby generating a frozen liquid substance portion 4 and a first evaporated liquid substance portion 5A

The first evaporated liquid substance portion 5A may be condensed by condensing means 6, whilst at the same time collecting the frozen liquid substance portion 4 as a layer 7 on a collection surface 8

The frozen liquid substance portion 4 collected as the layer 6 is conveyed along the collection surface 8 The surface 8 conveys the frozen liquid substance portion layer 7 at a rate which controls the thickness of the layer of collected frozen liquid substance portion

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collected

The condensed first evaporated liquid substance portion may be removed from the chamber 2 In order for any water or evaporated liquid substance portion to be removed from the gaseous phase in the chamber, it can be condensed on the coils In order for evaporated portion to be removed from the condensing coils, the coils can be returned to pressures above the triple point so that the ice will melt Therefore, in order to achieve substantially continuous or quasi-continuous operation two or more sets of condensation coils may be used, while one is frosting up, the other or remaining coils may be isolated from the drying chamber by closing a pressure seal of some sort, returning to ambient pressure, and defrosted

Liquid substance 3 in a holding tank T may be sprayed into the chamber 2 in order to achieve a pre-determined size of frozen liquid substance portion particles, and this may be performed by one or more nozzles 10

Thickness of the frozen liquid substance portion (particles) collected on the collection surface is determined by the rate (metres per second) of conveyance by the collection surface 8 which is in form of an endless conveyor (not shown) The thickness of this monolayer of particles will also be determined by the particle sizes generated by injection of the liquid substance into the chamber and/or of liquid substance injection by the nozzle(s) 10

Desirably the surface conveys the frozen liquid substance portion away from the point of liquid substance injection, or the position of the one or more nozzles, at a rate which substantially achieves about the monolayer thickness of frozen liquid substance portion upon the collection surface 8

The collected frozen liquid substance portion can then be exposed to a heating means 11 which may induce at least some sublimation and thereby generation of a second evaporated liquid substance portion 5B and a product 12

The second evaporated liquid substance portion 5B can also be condensed by the condensing means 6 and removed from the chamber 2

The product 12 (in a state advantageously containing less liquid than at the holding tank, T, state) may be removed from the collection surface 8 by a product removal means 13 then evacuated from the chamber 2 via the outlet port 14

In a further embodiment of the process substantially as described above, an apparatus may be configured to generate a product 12 which is drier or more concentrated, than that of the liquid substance 3 in the holding tank, T

A chamber 2 is held at a pressure at least below the triple-point pressure of the liquid substance by a pressure reduction means, such as a gas evacuation pump 15

One or more injection ports, such as nozzles 10, may be employed through which the liquid substance 3 can be sprayed Upon injection (or spaying) of the liquid substance 3 into the chamber 2, a frozen liquid substance portion 4 and a first evaporated liquid substance portion 5A may be generated

A collection surface 8 is provided to substantially collect the generated frozen liquid substance portion 4 of the liquid substance 3

A condensing means 6 may be employed to condense the first evaporated liquid substance portion 5A, and the condensate may be subsequently drained and removed from the chamber 2

An outlet port 14 from the chamber may be used to evacuate a drier or more concentrate form of the liquid substance 3, in the form of a product 12

The frozen liquid substance portion 4 can be accumulated as a layer 7 on the collecting surface and conveyed away from the one or more injection ports, towards the outlet port, at a rate which allows the accumulation of not more than substantially a monolayer thickness of frozen liquid substance portion

The first evaporated liquid substance portion 5A can be condensed by the condensing means 6 and removed from the chamber The condensing means 6 is a cooled condensation coil or coils. The condensing means is cooled with a refrigerant, supplied from the refrigeration unit 16

The one or more injection ports comprise a spray or an atomisation nozzle or nozzles The nozzle or nozzles substantially determine the size of the frozen liquid substance portion generated

The collection surface includes one or a combination of the following conveyors a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray The angled tray and/or vibrating tray include a reduced friction surface comprising polytetrafluroethylene

The layer of frozen liquid substance portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports which may substantially induce sublimation of the frozen liquid substance portion layer 7 upon the surface 8 to form a second evaporated liquid substance portion 5B and a product 12.

The heating means is one or a combination of the following energy sources infra-red lamps, halogen lamps, incandescent lamps, microwaves or ohmic heating of the surface 8 itself which may act directly upon the frozen liquid substance portion 4 The heating means may also operate upon the frozen particles indirectly via heat transfer from the surround internal chamber equipment, such as heat transfer through the collection surface 8, or via radiation or reflection of energy from the surrounding chamber walls

A product removal device can be employed to remove the product from the collection surface which may have stuck or adhered to the surface Such a product removal device may be a scraping means and/or a brushing means

A scraping means may substantially contact the surface 8 with the product 12 thereon and move the product off the surface toward the outlet port 14, whereas a brushing means may be in the form of a rotating brush or fixed brush which also substantially contacts the surface with the product thereon and moves product toward the outlet port Once the product is removed from the chamber it may then be packaged or further processed (such as a granulation stage, or vacuum packaging to maintain the quality of the product)

The liquid substance 3 may be supplied under pressure to the injection point 10 via a pump 17, with the liquid substance passing into the depressurised environment of the chamber 2 through a series of valves 18A

The frozen portion 5A of the sprayed or atomised injected liquid substance 3 is advantageously directed to land and be collected upon a surface 8 which then conveys the frozen portion 4 away from the injection nozzle(s) 10

The surface 8 may promotes travel of the frozen portion 4 away from the injection nozzle(s) by a either use of or a combination of conveyor surface options These surface options are provided which allow the surface collected frozen portion 4 to preferably not accumulate to greater than about a monolayer thickness of frozen portion 4 The thickness or layer 7 of the frozen portion 4 upon the surface 8 directly determines the rate of sublimation (drying) during exposure to the heating means 11

Alternatively, an optimal thickness for rapid sublimation (drying) of the frozen portion may be determined by actively measuring the thickness of the layer on the surface, for example using light reflection techniques. Once the thickness of the layer being sprayed onto the surface is determined, the conveyer rate of removal of particles away from the injection port(s) can be altered to match the liquid substance inlet spray rate, or to optimise the liquid substance throughput (and product production) The spray rate or throughput of liquid substance may also be altered to be sympathetic with the rate of conveying of particles along the surface

Once the frozen portion 4 is exposed to the heating means 11, sublimation is preferably induced thereby resulting in additional liquid removal from the now frozen liquid substance The substantially reduced moisture content substance, which may be referred to as the 'product' 12, can then be removed from the conveying surface The product can then be collected and removed from the chamber 2 via an outlet port 14, which includes one or more valves 18B (or air locks) to prevent loss of chamber vacuum

As the chamber 2 is held at a vacuum, a vacuum pump 15 is required to obtain desired pressures in the chamber, for example, the triple-point of water is 611 3 Pa (or 4 585 mmHg, or 0 0887 psi) The pressure of the chamber must never be higher than the triple-point pressure of water, however, depending on the product, certain temperature constraints must be met to avoid glass formation and freezing point depression associated with the freeze concentration effects that occur as the product dries Some products must be kept below their eutectic temperature while for others the glass transition temperature is critical Since the pressure in the vessel will determine the sublimation temperature and hence the product temperature, often the process will have to operate at pressures significantly below 611 3 Pa, for example, coffee -21°C and 94 Pa, fruit juices -30°C and

38 Pa Of course, it is recognised that this technology may be applied to most liquid substances containing particles in suspension or a material dissolved therein A person skilled in the art of thermodynamics would understand the relationship of pressure and temperature, and how the chamber conditions determine the quantity of flash evaporation (generation of the first evaporated liquid substance portion) and resulting temperature of the frozen liquid substance portion formed

The chamber conditions may be varied to optimise the level of throughput and/or level of freezing occurring in the chamber

As the liquid feed may be fed through a nozzle directly into the vacuum chamber, the nozzle and feed pressure are chosen such that a stable spray-jet is maintained In a sub-triple point environment, a material cannot exist in the liquid state, meaning that some of the liquid is forced to vaporise almost instantaneously, causing the remaining liquid to freeze Since the enthalpy of fusion duty does not need to be supplied by an external energy source, it is effectively "free" In addition, as a result of the same process, approximately one seventh of the sublimation duty is also free This means that for a given load of product, the spray freeze dryer will require at least 10% less energy than a conventional batch freeze dryer

The product is frozen into very fine particles, whose maximum size is limited by the surface tension and vapour pressure (typically 100μm or less) The frozen particles land on a heated tray or belt where it dries as moisture sublimes off and is condensed by the condensing coils as it is conveyed along the length of the dryer Freeze drying times may be estimated from the following equation (Fellows, 1997)

$$t_d = \frac{x^2 \rho \Delta M \lambda_s}{8 k_d \Delta T}$$

Where t_d = drying time (s), x = product layer thickness (m), k_d = thermal conductivity of dry product (W/mK), ρ = bulk density of product (kg/m³), ΔM = change in moisture content (dry basis), ΔT = temperature driving force (°C) and λ_s = enthalpy of sublimation (J/kg) This equation shows that the drying time is proportional to the square of the thickness of the layer of the product on the drying surface

The present invention of the second aspect can be referred to as a spray freeze drying process in a quasi-continuous process, and may be integrated into a continuous production line, which would reduce the labour requirements associated with its operation, and could also eliminate the risk of exposure or product contamination In addition, the need for a subsequent granulation stage is removed, since the dried product exits the dryer in powdered form

A polytetrafluroethylene (PTFE) coated surface such as a belt conveying system can be attached to a variable-speed drive, allowing the residence time of the product on the belt to be varied between about 1 and 12 minutes depending on exposure to the heating means

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required to sublime off a desired amount of moisture. The length of the belt may be about 2m and the belt speeds may be varied between about 0.17m/min and 2m/min The product can be sprayed through multiple nozzles, for example 4 nozzles in parallel, in order to distribute the product evenly across the width of the belt (which may be about 0 5m)

It is anticipated that the majority of particle sizes will be in the region of 10 to 100µm, and therefore, since it is likely that the product will adhere to the surface (or belt) even with the PTFE (polytetrafluroethylene) coating, a removal means such as a knife-edge, or brush or scraping means arrangement can be employed at the end of the conveyer ((or any position along the surface) If the scraper or knife-edge is not effective enough, a PTFE or other type of material brush can also be used to scrape the particles off The brush arrangement may cause aggregation of the particles, which may or may not be beneficial, depending on the down-stream handling intended for the particular product

The feed nozzle 10 can protrude directly into the vacuum chamber 2 The pressure within the vacuum chamber 2 is maintained below the triple point pressure of the liquid 3 and therefore as the liquid substance 3 exits the nozzle 10 some of the water is evaporated 5A instantaneously ("flashed") as a result of the pressure drop below the liquid's triple point pressure The evaporation process removes substantial amounts of heat from the droplet with the result that the remaining moisture in the product Is frozen Based on the relative magnitudes of the latent heats of vaporisation (≈2250 kJ/kg) and fusion (≈333 kJ/kg), 1 gram of evaporated moisture would freeze approximately 6 5 grams of remaining water 4 Remaining moisture in the frozen liquid substance portion 4 may optionally be removed by an subjecting it to a heating means or energy source such as an infra-red, halogen lamp or other energy source 11 within the vacuum chamber

Potential advantages of this type of arrangement would also include the significant reduction in external energy (electricity) requirement as almost no electricity is required for freezing the product as well as the associated reduction in the tray-heating electricity requirement, and the difficulties associated with the conveyance of the frozen product (caking, compacting, agglomeration, thawing/re-freezing) can be largely be removed

The spray freeze drier involves less equipment (and therefore a reduced capital cost) due to its mechanically simpler design, and there is the potential to have much greater control of the lateral distribution of the product on the drying tray (because the spray pattern may be controlled whereas before, the frozen product simply dropped onto the tray)

Some of the major advantages of spray-freeze drying over conventional batch freeze drying are the greatly reduced drying times (a matter of seconds compared to a matter of hours), minimises possibility of damage to heat-sensitive materials, about a 10% reduction in energy costs due to the novel freezing process, which is effectively free of cost, and the need for subsequent granulation stage, which is often costly and can result in product loss is substantially removed

By utilising this quasi-continuous process exposure to contaminants is avoided and the spray freeze dryer is able to be integrated into a continuous production line, and is much less labour intensive than conventional freeze driers

In further aspects of the present invention as broadly defined by the first, second and/or fifth aspects, there is provided a drying process and apparatus as illustrated by a flow diagram of the improved continuous spray-freeze-drying process is shown in Figures 2, 3, 4 and 5 A liquid substance such as coffee (not shown) is chilled from its initial temperature to some lower temperature in a chiller 21 This step is optional The liquid feed is directed under pressure via a pump (not shown), atomised through a high pressure nozzle (not shown) into a freezing chamber 22 Ideally, the particle size of the atomised liquid substance should be less than substantially 200-300 micron (μm) The feed is introduced into the freezing chamber 22 with a co-current flow of cold air (-20°C or less) and atomised and frozen to produce an atomised frozen substance (ALS). The ALS is quickly frozen below its eutectic temperature to prevent melting The frozen ALS is pneumatically conveyed by the cold air stream to the spray freezer to a separating cyclone 23, where frozen ALS exits the bottom of the cyclone 23 and the outlet air stream exits the top of the cyclone 23. The outlet air stream from the spray freezer must be below the eutectic temperature of the frozen ALS A fan or blower 24 blows the outlet air stream from the cyclone through a refrigeration system, in this instance an evaporator 25, where the air is cooled back down to its initial temperature before returning to the spray freezing chamber 22

The frozen product then falls by gravity into a termination chamber (not shown) attached to the bottom of the cyclone A vacuum or air lock 27 allows the product to pass from the bottom of the cyclone into the drying chamber 28 without interruption to the vacuum inside the drying chamber The termination chamber and air lock are cooled with cold air from the refrigeration system, in this instance a cooler 26 to prevent the frozen ALS from melting. The drying chamber is maintained at an absolute pressure of substantially 200-400 micro-meters of Hg with a vacuum pump 212 Frozen ALS falls onto an inclined pneumatic vibrating tray 29 which conveys drying product towards the discharge end of the dryer To effect sublimation and freeze drying, the tray is heated by a radiant and/or conduction heat source 210 placed underneath the tray 29 The surface temperature of the tray is controlled down its length to ensure complete drying without damaging the product (not shown) The dried ALS (product) exits through the discharge vacuum lock 213 Two condensers 211 are required to run the system continuously One condenser would be operating normally during drying, while the other would be isolated from the drying chamber and would be defrosting

An improved drying process for a liquid substance, according to the first aspect includes one or more of the steps of chilling the liquid substance from an initial temperature, atomising the liquid substance so that a predetermined particle size is achieved, freezing the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen

ALS into a drying chamber which is held under a vacuum, heating the frozen ALS so as to effect sublimation and freeze drying and then collecting the dried ALS from the chamber

A liquid substance may be defined to be any substance containing a liquid These may for example include, milk, coffee, liquor, pharmaceuticals, nutraceuticals, function food, agriceuticals, or any other substance which has a moisture content The amount of liquid in a substance need only be minimal to allow over a period of time degradation of the substance, therefore removal of any "free" liquid is desirable

The conveying of ALS to a drying chamber may be achieved by any suitable conveying methods/apparatus For example, these may include pneumatic methods, mechanical methods, electrical or gravity assisted conveying methods may also be suitable

In another embodiment of the first and/or second aspect preferably there is provided an improved drying process for a liquid substance which may include one or more of the steps of chilling the liquid substance from an initial temperature, atomising the liquid substance so that a predetermined particle size is achieved, freezing the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which is held under a vacuum, conveying the ALS through the drying chamber, and heating the frozen ALS via a temperature gradient while it travels through the drying chamber so that the temperature grading can effect sublimation and drying whilst preventing or minimising any heat damage occurring to the ALS as it passes through the chamber to a collection point of the dried ALS

The step of chilling a liquid substance can take place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature. For example, this may mean that the liquid substance is chilled from ambient conditions and/or a storage condition facility temperature to a lower temperature This chilling step is optional, however it may assist and reduce the cooling loading required by the spray freezer

The chilling may be achieved by any suitable method or apparatus that can reduce the temperature of a substance from an initial temperature to a lower temperature. Preferably chilling may be achieved by any suitable heat transfer unit, these may for example be refrigerators, plate heat exchangers, shell and tube heat exchangers, heat pumps, cool air convection apparatus, gas-liquid cooling towards and other such suitable cooling apparatus

The spray freezer is an especially important aspect of the continuous processing configuration in which rapid temperature reduction to below the substance eutectic temperature is desired, as well as atomisation of the liquid substance Preferably, atomisation of the liquid substance may be induced by a variety of feed devices such as, single-liquid nozzle (pressure type), two-liquid nozzle (pneumatic type), centrifugal (spinning disc), ultrasonic nozzles and various other rotary atomisers and air atomisation techniques may be employed

The cooling of the ALS may be achieved by the use of a cooler capable of freezing the ALS Preferably the atomisation and cooling of the liquid substance both take place in a spray freezer.

There are a number of advantages to spray freezing, especially as particulates formed may be produced of specific or predetermined particle sizes and the specification of, or particulate quality, remains substantially constant throughout the entire spray freezer operation Spray freezer operation is ideally continuous and adaptable for full automatic control, where response times are fast Spray freezing is a useful application to both heat sensitive and heat resistant substances. However, if is appreciated that semi-continuous processing may also be required

A phase change occurs in the ALS when the temperature of the ALS is reduced, that is in which liquids become frozen or reduced to below their eutectic temperature (i e solids) Liquids may be sublimated from the frozen ALS at a later state in the process once exposed to a temperature gradient and energy source

Cyclones are one of the main methods used to separate gas-solid phases, and can provide efficient separation Frozen ALS and gases are separated, with the gas being recycled to chill the liquid substance, and the frozen (solid) ALS being conveyed through the drying chamber

Sublimation is the phase change from frozen liquids (solid) to gas (liquid vapour) for the means of evaporation of liquid from the ALS Ideally the sublimation and/or drying of the frozen ALS takes place on a surface More preferably the surface may be a vibrating surface Even more preferably, the vibrating surface is a vibrating tray in which the vibrations are produced by pneumatic and/or mechanical and/or electrical means It is advantageous to induce and/or provide some vibration movement of the frozen ALS to be dried to enhance and promote improved heat transfer to the atomised particles

The sublimation (i e drying of the ALS and "free" liquid removal) may be initiated and effected by the energy emitted from an energy source such as an infrared device, which the vibrating drying tray preferably enhances the heat transfer characteristics to the ALS and allows for a more uniform and controlled drying stage The energy source capable of heating the ALS may be any suitable energy source for inducing sublimation of the frozen liquids of the ALS Such suitable energy sources may for example include, infra-red emitting devices, microwaves, radiant heaters, convection heaters, and any device which provides enough energy to the ALS to induce sublimation of the frozen "free" liquids of the ALS

The removal of liquids from substances to form a dried product by controlling air flow rates, temperature and pressure reduced the moisture content of substances assists in inhibiting microbial growth that may cause decay and spoilage Moisture removal also reduces weight, which is of significant interest and consideration for shipping and transport

Chemical pre-treatment of some substances may further aid the longevity of product shelf life These pre-treatments may include substances to enhance preservation

Often dehydration on an industrial scale requires a feed product to be reduced in size and/or particulated to allow enhanced processing characteristics Freeze drying is the drying of material in a frozen state and preferably the drying stage takes places at an absolute pressure that may readily permit ice to undergo a phase change directly from solid to vapour In the present invention freeze dried product is preferably substantially undamaged from the drying chamber stage

In an improved drying process of the first and second aspects, the liquid substance may be atomised in a spray freezer to achieve a particle size of substantially 500 microns or more Alternatively, the particle size achieved may be less than substantially 500 microns, or even less than substantially 200 microns Atomisation of the liquid substance is desirable so that the atomised liquid substance may be rapidly reduced in temperature to below its eutectic temperature Further, atomisation of the liquid substance and subsequent freezing allows for a particulate product to be processed which allows for enhanced drying control and heat transfer characteristics

The apparatus or spray freezer may be of a counter current configuration, although it may also be of co-current configuration in which the spray freezer can utilise a cold gas to effect a phase change of the liquids in the ALS. The cold gas can effect a phase change causes freezing of liquids within the atomised liquid substance (that is, the ALS is reduced to a temperature below its eutectic temperature) The cold gas utilised by the spray freezer to effect the phase change may be cold air, although any suitable gas to effect a phase change of liquids contained within the ALS may be used

The cold gas used may be substantially 0°C or less, even more preferably the cold gas may be less than substantially - 20°C

The ALS once frozen/reduced to below its eutectic temperature can then be conveyed to a separator, and conveying may take place pneumatically The separator can be a gas-solid separation device, for instance a cyclone

The gas separated from the cyclone may be returned and/or refrigerated for use in the spray freezer It can also be recycled to the spray freezer to reduce the cooling load on the evaporator The evaporator is used to cool the gas which is used to induce a phase change of liquids in the ALS However, any suitable gas cooling apparatus may be used, with an evaporator being used as an example in this case Suitable cooling apparatus may include refrigerators, plate heat exchangers, shell and tube heat exchangers and other heat transfer units capable of cooling a gas to a temperature that can be supplied to a spray freezer to force a phase change of liquids in the ALS to the solid (frozen) state.

Preferably one or more vacuum and/or air locks are in place between the frozen ALS cyclone outlet and drying chamber inlet, or some other type of pressure seal device It is advantageous to provide one or more pressure lock devices for maintenance of the vacuum within the drying chamber Without the use of these pressure lock devices a suitable vacuum in the drying chamber may not be obtainable Pressure locks such as valves may be particularly suitable, although any suitable device for sealing the vacuum drying chamber and ensuring the drying chamber maintains its vacuum may be utilised

In further embodiments there can be a refrigeration system which is employed to maintain the frozen ALS at a temperature below its eutectic temperature during transport from the cyclone to the vacuumous drying chamber. It is advantageous that the frozen ALS remains at a temperature below it eutectic temperature to minimise any product degradation or deterioration that may occur if the ALS rises about its eutectic temperature

The vacuum of the vacuumous drying chamber is created by a suitable pressure reduction device

The vacuum created by such suitable pressure reduction devices may be substantially 600 micron meters Hg at select pressure or less More preferably the vacuum created may be in the range of substantially 200-400 micro meters Hg absolute pressure

Transport or conveying of the frozen ALS through the vacuum chamber can take place on a vibrating surface, where the vibrating surface is a vibrating tray The vibrating tray may be pneumatically, electrically and/or via mechanical means driven for provision of the vibration to the tray

The vibrating tray conveys frozen ALS through a temperature gradient to effect sublimation, with the vapour produced by sublimation being removed from the drying chamber by one or more condensers or such like.

The temperature gradient across the drying tray is provided by an energy source, such as an infrared emitting device However, the use of other energy sources are envisaged, for example Ohmic heaters and micro-wave devices

The product produced by an improved drying process and apparatus is substantially free of liquid, otherwise substantially dried or at least substantially reduced in liquid content

An apparatus for improved drying which facilitates one or more of the following steps of chilling a liquid substance from an initial temperature to a lower temperature, atomising a liquid substance so that a predetermined particle size may be achieved, cooling the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which may be held under a vacuum, and heating the frozen ALS so as to effect sublimation and freeze drying and then collecting the dried ALS from the drying chamber is desired In an alternative embodiment the apparatus is defined as an apparatus for an improved drying process which may facilitate one or more of the steps of chilling the liquid substance from an initial temperature, atomising the liquid substance so that a predetermined particle size may be achieved, cooling the atomised liquid substance (ALS) to below its eutectic temperature, conveying the frozen ALS into a drying chamber which is held under a vacuum, conveying the ALS through the drying chamber and, heating the frozen ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation and drying whilst preventing or minimising any heat damage occurring to the ALS as it passes through the chamber to a collection point of the dried ALS

It would be preferable but not necessarily essential that the steps of the process and methodology may be linked to form a continuous process A continuous process may have a number of advantages

In addition, a process which is configured to be run on a continuous basis may be preferable as a continuous system tends to be more easily optimised for productivity, higher throughput, reduced processing time per unit produced, improved quality as a result of optimised controls, a generally more efficient process, reduction of human handling and/or contact with substances which may contaminate equipment and/or substances is desirable, and the reduction of human manual labour preferably also reduce the likelihood of injury Further, continuous processing may allow for equipment designed for an optimised solution for continuous processing and production of freeze dried products

The vacuum drying chamber is designed to operate at absolute pressures in the range of 200-400 microns of Hg The vacuumous state of the drying chamber allows for the preferential phase change (sublimation) of the frozen liquids to gas/vapour phase The sublimation material (vapour) may be preferably removed by the use of a condenser In a preferred embodiment the drying chamber is run continuously, and may be linked with additional spray freezers, separating devices (cyclones), packaging systems to be run in a batch, semi-continuous or continuous basis

In a preferred embodiment of the present invention, the frozen ALS may fall onto an inclined pneumatic vibrating tray, (although other vibrating tray systems may be employed) to convey the ALS through a temperature gradient to effect sublimation and discharge of dried product at the end of the drier The dried product exits through a discharge vacuum lock (which ensures the vacuum is maintained) into further processing stages For example, further processing stages may include discrete packaging systems

It is preferable that the surface temperature of the tray is controlled down its length to ensure complete drying without damaging of products

Drying may be defined as the application of energy under controlled conditions to remove the majority of the water normally present in a substance by evaporation The main purpose of dehydration is to extend the shelf life of substances by a reduction in water content

Drying may cause a deterioration in the eating quality and nutritive value of the food Water plays an important role in the stability of fresh, frozen and dried foods as it acts as a solvent for chemical, microbiological and enzymatic reaction

A dryer may include a vibrating tray, a pressure reducing means, a material inlet port, a material outlet port, and a heat source The vibrating tray provides a vibrating surface (upon which sublimation occurs) which conveys frozen ALS from the inlet material port to

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the outlet material port The inlet material port is the entry point for frozen ALS into the dryer, and the outlet material port is the exit point of dried ALS (post sublimation)

A heat source is used to induce sublimation of frozen liquids from the frozen ALS along the length of the vibrating tray

It is therefore advantageous to provide a series of treatment stages in which a liquid substance may be processed and substantially dried or have its liquid content reduced

The steps as described of optionally chilling a liquid substance, atomising the liquid substance, cooling the ALS to initiate a phase change of the liquids within the ALS, optionally separating the phase changed ALS from the cooling gas, conveying the phase changed ALS to a drying chamber, and removal of liquids from the ALS by sublimation may be referred to as a series of treatment stages to provide a substantially reduced liquid/moisture content substance

However, it is also considered that the pre-treatment steps may be foregone and the liquid substance may be inserted directly to the vacuum chamber (which is held at a vacuum pressure which may be below the triple-point pressure of the liquid substance being processed) This direct injection may result in the flash evaporation of a portion of the liquid with the remainder of the liquid substance consequently being reduced in temperature such that frozen liquid substance particles result These frozen particles may then be treated within the drying chamber to induce sublimation of remaining liquid in order to achieve a substantially drier liquid substance

The pre-treatment stages may be considered to enhance the overall process, although they may be foregone where direct injection of the liquid substance to the vacuum pressure drying chamber is an option

Those skilled in the art will appreciate, understand and be able to calculate the sizings of pumps, heaters, motors, spray or atomisation nozzles required to achieve the present invention, and it is appreciated that there are numerous variables which will dictate or influence the sizings of these sorts of components It should also be appreciated that design of a vacuum chamber will be within the realm of a person skilled in the art – however it is the combination and advantages conveyed above that make this invention suitable

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope thereof as defined in the appended claims

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THE CLAIMS DEFINING THE INVENTION ARE:

1 A method of drying or concentrating a liquid substance having solid particles in suspension or a substance dissolved therein including the steps of
holding a chamber at a temperature and pressure below the triple-point of the liquid substance,
injecting and/or atomising the liquid substance into the chamber to generate a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),
collecting the FLS portion as a layer on a surface, and
conveying the collected FLS portion on the surface,
wherein the surface conveys the collected FLS portion at a rate which controls the thickness of the layer of collected FLS on the surface

2 A method as claimed in claim 1, wherein the rate of conveying achieves substantially a monolayer thickness of FLS portion to accumulate on said surface

3 A method as claimed in claim 1 or claim 2, wherein the FEL is condensed by a condensing device.

4 A method as claimed in anyone of claims 1 to 3, wherein the FEL is removed from said chamber

5 A method as claimed in any one of the preceding claims, wherein injecting and/or atomising the liquid substance includes spraying to achieve a pre-determined particle size of FLS

6. A method as claimed in any one of the preceding claims, wherein injecting and/or atomising of the liquid substance is performed by one or more nozzles.

7 A method as claimed in any one of the preceding claims, wherein the rate of conveyance of FLS upon said surface achieves substantially a monolayer thickness of the FLS upon said surface

8 A method as claimed in claim 7, wherein the monolayer may be the thickness of a single layer of the pre-determined particle size of the FLS portion.

9 A method as claimed in claim 8, wherein the predetermined particle size is substantially 500 μm or more

10 A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 500 μm

11 A method as claimed in claim 8, wherein the predetermined particle size is less than substantially 200 μm

12 A method as claimed in any one of the preceding claims, wherein the surface conveys the FLS portion away from the one or more nozzles.

13. A method as claimed in any one of the preceding claims, wherein the method

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includes the step of exposing the collected layer of FLS portion to a heating means thereby substantially inducing sublimation and generation of a second evaporated liquid substance portion ("SEL portion") and a product.

14 A method as claimed in claim 13, wherein the SEL portion is condensed by a condensing device

15 A method as claimed in claim 13 or claim 14, wherein the SEL portion is removed from the chamber

16 A method as claimed in any one of claims 13 to 15, wherein the product is removed from the conveying surface by a product removal device

17 A method as claimed in any one of claims 13 to 16, wherein the product is removed from the chamber via the outlet port.

18. A method as claimed in any one of the preceding claims, wherein the method is used to process a fluidised substance, or a liquid substance having solid particles in suspension or a substance dissolved therein

19 A method as claimed in any one of the preceding claims where the substance is selected from one or more of the following. a slurry of coffee, liquid milk, fruit and/or vegetable juices

20 An apparatus for drying or concentrating a liquid substance comprising.

a chamber and a chamber pressure reduction device, and

one or more injection ports through which the liquid substance is injected to the chamber,

a collection surface which collects a frozen liquid substance portion of the liquid substance,

wherein the pressure reduction device maintains the chamber at a pressure below at least the triple-point pressure of the liquid substance, to cause the injected liquid substance to separate into a frozen liquid substance portion ("FLS portion") and a first evaporated liquid substance portion ("FEL portion"),

so that in use, the FLS portion is accumulated as a layer on the collecting surface and conveyed away from the one or more injection ports at a rate which allows the thickness of the layer of the FLS portion to be controlled.

21 An apparatus as claimed in claim 20, wherein the rate of conveyance achieves substantially a monolayer thickness of FLS portion to accumulate on said surface.

22 An apparatus as claimed in claim 20 or claim 21, wherein the FEL portion is condensed by a condensing device

23 An apparatus as claimed in any one of claims 20 to 22, wherein said FEL is removed from said chamber.

24 An apparatus as claimed in any one claims 20 to claim 23, wherein the one or more injection ports comprises at least one spray or atomisation nozzle.

25. An apparatus as claimed in claim 24, wherein the nozzle or nozzles substantially determine the size of the FLS portion generated to achieve a pre-determined particle size

26 An apparatus as claimed in any one of claims 20 to 25, wherein the pressure reduction device is a gas evacuation pump

27 An apparatus as claimed in any one of claims 20 to 26, wherein the condensing device is a cooled coil or coils

28 An apparatus as claimed in any one of claims 20 to 27, wherein the condensing device is cooled with a refrigerant

29 An apparatus as claimed in any one of claims 20 to 28, wherein the collection surface includes one, or a combination of, the following conveyors a moving endless belt configuration, a tray angled to encourage the frozen liquid substance portion to slide away from the one or more injection ports, a vibrating tray

30 An apparatus as claimed in claim 29, wherein the angled tray and/or vibrating tray includes a reduced friction surface.

31. An apparatus as claimed in claim 30, wherein the reduced friction surface comprises polytetrafluroethylene (PTFE).

32 An apparatus as claimed in any one of claims 20 to 31, wherein the layer of FLS portion upon said collection surface is exposed to a heating means as it is conveyed away from the one or more injection ports

33 An apparatus as claimed in claim 32, wherein the heating means substantially induces sublimation of the FLS portion layer upon said surface to form a second evaporated liquid ("SEL") substance portion and a product

34 An apparatus as claimed in claim 32 or claim 33, wherein the heating means is one or a combination of the following energy sources. infra-red lamps, halogen lamps, incandescent lamps, microwaves, or ohmic heating of said surface.

35 An apparatus as claimed in claim 33 or claim 34, wherein a product removal device is employed to remove the product from the collection surface.

36 An apparatus as claimed in claim 35, wherein the product removal device is a scraping means and/or a brushing means.

37 An apparatus as claimed in claim 36, wherein the scraping means substantially contacts the surface with the product thereon and moves the product off the surface to the outlet port.

38. An apparatus as claimed in claim 36, wherein the brushing means is a rotating brush or fixed brush which substantially contacts the surface with the product thereon and channels the product off the surface to the outlet port.

39. An apparatus as claimed in any one of claims 33 to 37, wherein the product is removed via a chamber outlet port

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40 An apparatus as claimed in any one of claims 20 to 39, wherein the chamber is held at a pressure of substantially 611 3 Pa or less

41 A drying process for a liquid substance comprising the steps of.
atomising the liquid substance,
cooling the atomised liquid substance (ALS) to initiate a phase change,
conveying the ALS into a drying chamber under a vacuum,
heating the ALS so as to substantially effect sublimation and then collecting the substantially dried ALS from the chamber

42 A drying process as claimed in claim 41, wherein the liquid substance is chilled from an initial temperature prior to atomising.

43 A drying process as claimed in claim 41 or claim 42, wherein the ALS conveyed into the drying chamber is also conveyed through the drying chamber

44. A drying process as claimed in any one of claims 41 to 43, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation

45 A drying process as claimed in any one of claims 42 to 44, wherein the step of chilling a liquid substance takes place in a chiller to reduce the liquid substance from an initial temperature to a lower temperature

46. A drying process as claimed in any one of claims 41 to 45, wherein atomising the liquid substance achieves a predetermined particle size

47 A drying process as claimed in claim 46, wherein the predetermined particle size is substantially 500 μm or more

48 A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 500 μm

49 A drying process as claimed in claim 46, wherein the predetermined particle size is less than substantially 200 μm

50 A drying process as claimed in any one of claims 41 to 49, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS)

51 A drying process as claimed in any one of claims 42 to 50, wherein chilling is undertaken with direct or indirect contact with a cold fluid.

52 A drying process as claimed in claim 51, wherein said cold fluid is air

53 A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is substantially 0°C or less.

54 A drying process as claimed in claim 51 or claim 52, wherein said cold fluid is less than substantially -20°C

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55 A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a counter current configuration.

56 A drying process as claimed in any one of claims 50 to 54, wherein said spray freezer operates in a co-current configuration

57 A drying process as claimed in any one of claims 41 to 56, wherein the ALS is reduced to a temperature below its eutectic temperature

58 A drying process as claimed in any one of claims 41 to 57, wherein the ALS is conveyed to a separator.

59. A drying process as claimed in any one of claims 41 to 57, wherein said ALS is conveyed pneumatically to a separator

60. A drying process as claimed in claim 58 or claim 59, wherein said separator is a gas-solid separation device.

61 A drying process as claimed in claim 60, wherein said gas-solid separation device is a cyclone.

62. A drying process as claimed in claim 60 or claim 61, wherein the gas separated in the gas-solid separation device is returned and/or refrigerated for use in the chilling step

63 A drying process as claimed in claim 61 or claim 62, wherein one or more vacuum and/or air locks are present between a AFS cyclone outlet and a drying chamber inlet

64 A drying process as claimed in any one of claims 60 to 63, wherein separated solids from said gas-solid separation device enter a vacuumous drying chamber.

65 A drying process as claimed in any one of claims 41 to 64, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device

66. A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less

67 A drying process as claimed in claim 65, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure

68 A drying process as claimed in any one of claims 41 to 67, wherein said ALS is conveyed via a surface

69 A drying process as claimed in any one of claims 41 to 68, wherein said ALS is conveyed via a vibrating surface.

70 A drying process as claimed in claim 69, wherein said vibrating surface is a vibrating tray.

71. A drying process as claimed in claim 69 or claim 70, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

72 A drying process as claimed in claim 70 or claim 71, wherein said vibrating tray conveys ALS through a temperature gradient to substantially effect sublimation 210

73 A drying process as claimed in any one of claims 41 to 72, wherein a vapour produced by sublimation is removed from the drying chamber.

74 A drying process as claimed in claim 73, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers

75 A drying process as claimed in any one of claims 41 to 74, wherein the ALS is heated via a temperature gradient while it travels through the drying chamber so that the temperature gradient substantially effects sublimation.

76 A drying process as claimed in any one of claims 41 to 75, wherein said temperature gradient is provided by an energy source

77 A drying process as claimed in claim 76, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater

78 A drying process as claimed in any one of claims 41 to 77, wherein atomising the liquid substance achieves a predetermined particle size.

79 A drying process as claimed in claim 78, wherein the predetermined particle size is substantially 500 μm or more.

80 A drying process as claimed in claim 78, wherein the predetermined particle size is less than substantially 500 μm.

81. A drying process as claim in claim 78, wherein the predetermined particle size is less than substantially 200 μm

82 A drying process as claimed in any one of claims 60 to 81, wherein a refrigeration system is employed to maintain ALS at a temperature below its eutectic temperature during transport from the gas-solid separation device to the vacuumous drying chamber.

83 A drying process as claimed in any one of claims 41 to 82, wherein a product produced is substantially reduced is free of liquid compared to the liquid substance.

84 An apparatus for a drying process for a liquid substance comprising.

an atomiser capable of atomising a liquid substance,

a cooler capable of cooling the atomised liquid substance (ALS) to initiate a phase change,

a conveyor capable of conveying the ALS into a drying chamber held under a vacuum,

an energy source capable of heating the ALS so as to effect sublimation and freeze drying, and

a collector capable of collecting the dried ALS

85 An apparatus for a drying a drying process as claimed in claim 84, wherein a chiller capable of chilling a liquid substance from an initial temperature to a lower

temperature is provided prior to the liquid substance atomiser

86 An apparatus for a drying a drying process as claimed in claim 84 or claim 85, wherein the apparatus includes a conveyor capable of conveying the ALS through the drying chamber

87 An apparatus for a drying a drying process as claimed in any one of claims 84 to 86, wherein the apparatus includes an energy source capable of heating the ALS via a temperature gradient while it travels through the drying chamber so that the temperature gradient can effect sublimation.

88 An apparatus for a drying process as claimed in claim 87, wherein the temperature gradient substantially prevents or substantially minimises heat damage occurring to the ALS as it passes through the chamber

89 An apparatus for a drying process as claimed in any one of claims 84 to 88, wherein atomising the liquid substance achieves a predetermined particle size

90 An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is substantially 500 μm or more

91 An apparatus for a drying process as claimed in claim 89, wherein the predetermined particle size is less than substantially 500 μm

92 An apparatus for a drying process as claimed in claim 89, wherein the predetermined size is less than substantially 200 μm

93 An apparatus for a drying process as claimed in any one of claims 84 to 92, wherein a spray freezer utilises a cold gas to effect a phase change of the liquids in the atomised liquid substance (ALS).

94 An apparatus for a drying process as claimed in claim 93, wherein said cold gas is air

95 An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is substantially 0°C or less

96 An apparatus for a drying process as claimed in claim 93 or claim 94, wherein said cold gas is less than substantially -20°C

97 An apparatus for a drying process as claimed in any one of claims 84 to 96, wherein the vacuum of said vacuumous drying chamber is created by a pressure reduction device

98. An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is substantially 600 micro meters Hg absolute pressure or less

99 An apparatus for a drying process as claimed in claim 97, wherein the vacuum created by the pressure reduction device is in the range of substantially 200-400 micro meters Hg absolute pressure.

100 An apparatus for a drying process as claimed in any one of claims 84 to 99, wherein said ALS is conveyed via a vibrating surface.

101. An apparatus for a drying process as claimed in claim 100, wherein said vibrating surface is a vibrating tray.

102 An apparatus for a drying process as claimed in claim 101, wherein said vibrating tray is pneumatically and/or mechanically and/or electrically and/or magnetically driven

103 An apparatus for a drying process as claimed in claim 84 or claim 102, wherein ALS is conveyed through a temperature gradient to substantially effect sublimation.

104 An apparatus for a drying process as claimed in claim 84 or claim 103, wherein vapour produced by sublimation is removed from the drying chamber by one or more condensers.

105. An apparatus for a drying process as claimed in claim 103, wherein said temperature gradient is provided by an energy source

106 An apparatus for a drying process as claimed in claim 105, wherein said energy source is an infra-red emitting device and/or a micro-wave emitting device and/or an ohmic heater

107 A drying chamber for use with the apparatus as claimed in any one of claims 20 to 40 which comprises

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray

108 A drying chamber for use with the apparatus as claimed in anyone of claims 84 to 106 which comprises·

a device capable of vibrating a tray,

a suitable device for reducing the pressure in said chamber,

a material outlet port from said chamber, and

a heat source adapted to act upon said tray

ABSTRACT

This invention relates to a process and/or method and associated apparatus for use in spray freeze drying a fluid substance such as fruit juice, pharmaceuticals, nutraceuticals, tea and coffee A fluid substance is sprayed (atomised) and reduced in temperature to below its eutectic temperature; thereby inducing a phase change of fluids in the fluid substance The atomised fluid substance (AFS) is then subjected to a vacuumous drying chamber in which the AFS is subjected to an energy source to assist sublimation of fluid from the AFS as it is conveyed through the chamber. Such an energy source can provide a temperature gradient through which the AFS passes and is exposed to, all carried out so as to preferably minimise or reduce the likelihood of degradation of the substance being dried (reducing the fluid content).

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AS: 102.362 [redacted]

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SUPERINTENDENCIA

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Eve 379 FASENACIONALI
Act 411 PRESENTACION Folios 213

FORMULARIO ÚNICO DE SOLICITUD DE PATENTE
PCT
ENTRADA EN FASE NACIONAL

1 SOLICITUD DE:

☒ Patente de Invención. ☐ Patente de Modelo de Utilidad.

☐ Capítulo I. ☒ Capítulo II.

2 **SOLICITANTE (71)**	Nombre: **AGRESEARCH LIMITED** Dirección East Street, Ruakura Campus, Hamilton, Private Bag 3123, 2020 Hamilton, Nueva Zelanda Nacionalidad o Domicilio: Nueva Zelanda Lugar de Constitución: Nueva Zelanda Teléfono: Fax E-mail	**IDENTIFICACION** C.C. ☐ NIT ☐ C.E. ☐ Otro ☐ Cual ______ Número ______
3 **REPRESENTANTE O APODERADO**	Nombre: JUAN PABLO CADENA SARMIENTO Dirección: Cra. 7 No. 71-21, Torre B, Piso 4 Teléfono: 744-2200 Fax: 742-2200 E-mail:	**IDENTIFICACION** C.C. ☒ NIT ☐ C.E. ☐ Otro ☐ Cual ______ Número 80.410.337 TP 57492
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5 PCT (86)

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6 Título (54): PROCESO Y APARATO DE SECADO

7 Clasificación Internacional (51) ______

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9 Comprobante de pago No. 06-74.632 Fecha 01-11-2006

Instrucciones para el diligenciamiento del presente formulario, ver reverso de esta página

[Continued on next page]

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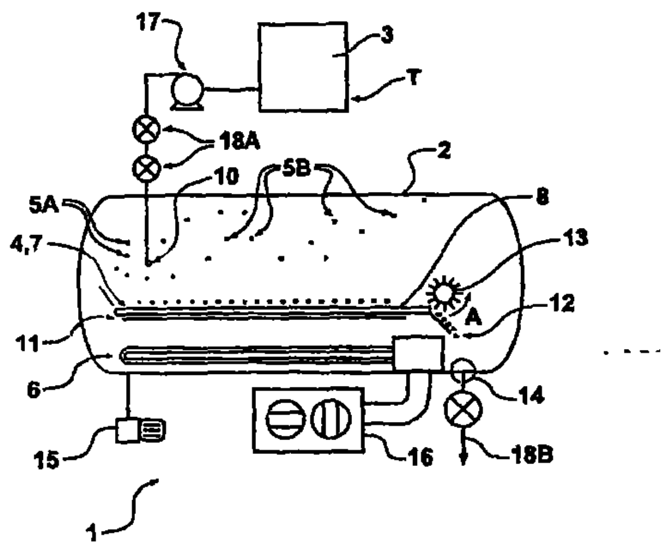
(54) Title: DRYING PROCESS AND APPARATUS



(57) Abstract: This invention relates to a process and/or method and associated apparatus for use in spray freeze drying a liquid substance such as fruit juice, pharmaceuticals, nutraceuticals, tea and coffee. A liquid substance is sprayed (atomised) and reduced in temperature to below its eutectic temperature, thereby inducing a phase change of liquids in the liquid substance The atomised liquid substance (ALS) is then subjected to a vacuumous drying chamber in which the ALS is subjected to an energy source to assist sublimation of liquid from the ALS as it is conveyed through the chamber. Such an energy source can provide a temperature gradient through which the ALS passes and is exposed to, all carried out so as to preferably minimise or reduce the likelihood of degradation of the substance being dried (reducing the liquid content)

WO 2005/105253 A1

LAS REIVINDICACIONES QUE DEFINEN LA INVENCIÓN SON:

1. Un método de secar o concentrar una sustancia líquida que tiene partículas sólidas en suspensión o una sustancia disuelta en ella, el cual incluye los pasos de:
mantener una cámara a una temperatura y presión por debajo del punto triple de la sustancia líquida,
inyectar y/o atomizar la sustancia líquida a la cámara para generar una porción de la sustancia líquida congelada ("porción FLS") y una primera porción de la sustancia líquida evaporada ("porción FEL"),
recoger la porción FLS como una capa en una superficie, y
conducir la porción FLS recogida en la superficie,
en donde la superficie conduce la porción FLS recogida a una velocidad que controla el espesor de la capa de FLS recogida en la superficie.

2. Un método como se reivindica en la reivindicación 1, en donde la velocidad de conducción logra sustancialmente que un espesor de monocapa de la porción FLS se acumule en dicha superficie.

3. Un método como se reivindica en la reivindicación 1 o la reivindicación 2, en donde la FEL se condensa por un dispositivo condensador.

4. Un método como se reivindica en una cualquiera de las reivindicaciones 1 a 3, en donde la FEL se extrae de dicha cámara.

5. Un método como se reivindica en una cualquiera de las reivindicaciones precedentes, en donde inyectar y/o atomizar la sustancia líquida incluye atomización para obtener un tamaño de partícula predeterminado de FLS.

6. Un método

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Fec 2006-11-01 16 27 24 Dep 2020 NUECREACIONES
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Eve 379 FASENACIONALII
Act 411 PRESENTACION Folios 213

REQUISITOS MINIMOS PARA ADMISION A TRAMITE PCT

PATENTE DE INVENCION [x] MODELO DE UTILIDAD [ ]

- Indicación de que se solicita la concesión de una patente [x]
- Datos de identificación del solicitante o de la persona que presenta la solicitud [x]
- Descripción de la invención [x]
- Dibujos de ser estos pertinentes [x]
- Comprobante de pago de las tasas establecidas [x]

COMPLETA [x] INCOMPLETA [ ]

DISEÑO INDUSTRIAL [ ]

- Indicación de que se solicita el registro de Diseño Industrial [ ]
- Datos de identificación del solicitante o de la persona que presenta la solicitud [ ]
- Representación gráfica y fotográfica del Diseño Industrial o muestra del material que incorpora el diseño [ ]
- Comprobante de pago de las tasas establecidas [ ]

COMPLETA [ ] INCOMPLETA [ ]

ESQUEMA DE TRAZADO [ ]

- Indicación de que se solicita el registro de un Esquema de Trazado [ ]
- Datos de identificación del solicitante o de la persona que presenta la solicitud [ ]
- Representación gráfica del esquema trazado [ ]
- Comprobante de pago de las tasas establecidas [ ]

COMPLETA [ ] INCOMPLETA [ ]

Fdo B
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Fecha 01-NOV-6

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REPUBLICA DE COLOMBIA
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

Bogotá,
2020

Doctor(a)
CADENA SARMIENTO JUAN PABLO

REFERENCIA	Radicación No.	6 111319
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	Fecha inicio fase nacional	01/12/2006
	Trámite	11
	Evento	379
	Actuación	430
	Oficio No.	14292

Como resultado del examen de forma, realizado con fundamento en el artículo 38 de la Decisión 486 de la Comisión de la Comunidad Andina, artículo 27 del Tratado de Cooperación en Materia de Patentes (PCT), regla 51 bis del Reglamento y Circular Única de la Superintendencia de Industria y Comercio, se le comunica que:

1. La solicitud no contiene la copia del documento en que consta la cesión del derecho a la patente del inventor al solicitante o a su causante. (Art. 26-k) Decisión 486.

2. La solicitud no contiene el poder debidamente otorgado, con el lleno de los requisitos exigidos por los artículos 63 y subsiguientes del Código de Procedimiento Civil. Adicionalmente, deberá presentar el documento que acredita la existencia y representación legal de la sociedad solicitante, cuando ésta fuere persona jurídica.

En cumplimiento de lo dispuesto por el artículo 39 de la Decisión 486, se le requiere para que dentro del término de dos meses siguientes a la fecha de notificación del presente oficio, complete los requisitos anteriormente enunciados. En caso de no dar cumplimiento al presente requerimiento, la solicitud se considerará abandonada y perderá su prelación.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo quinto del título primero de la Circular Unica No.10 de 2001.

ALIX CARMENZA CESPEDES DE VERGEL
Jefe de la división de Nuevas Creaciones

El anterior requerimiento fue notificado por fijación en lista de fecha 11 DIC. 2006
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