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EP 0749770 A1 **US 4162926 A**
US 3956009 A

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(54) Abstract Title
Spray-drying a material in the presence of a particulate solid

(57) A spray-drying process comprises spray-drying a first material dispersed in a solvent at a concentration of 1-50% w/v in the presence of a second finely divided particulate solid material which is continuously introduced into the spray dryer in an amount from 10-99% w/w of the final product. The product is a matrix of the first material bonded to the particles of the second. The first material may comprise a gum, protein or sugar e.g. sucrose, maltodextrin, lactose, molasses and it may also comprise colourings/flavourings. The inlet temperature of the air may be 140-220°C. The second material may comprise crystalline particles e.g. sugar and /or it may be the same as the first material. The mean particle size of the second material may be 1-450 µm. A free-flowing particulate material is obtainable by such a process which may comprise an amorphous matrix of first material bonded to the second and also spheroidal particles of the first. The free-flowing material may be directly compressible and non-hygroscopic and it may be made into a tablet by compression.

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FIG. 1

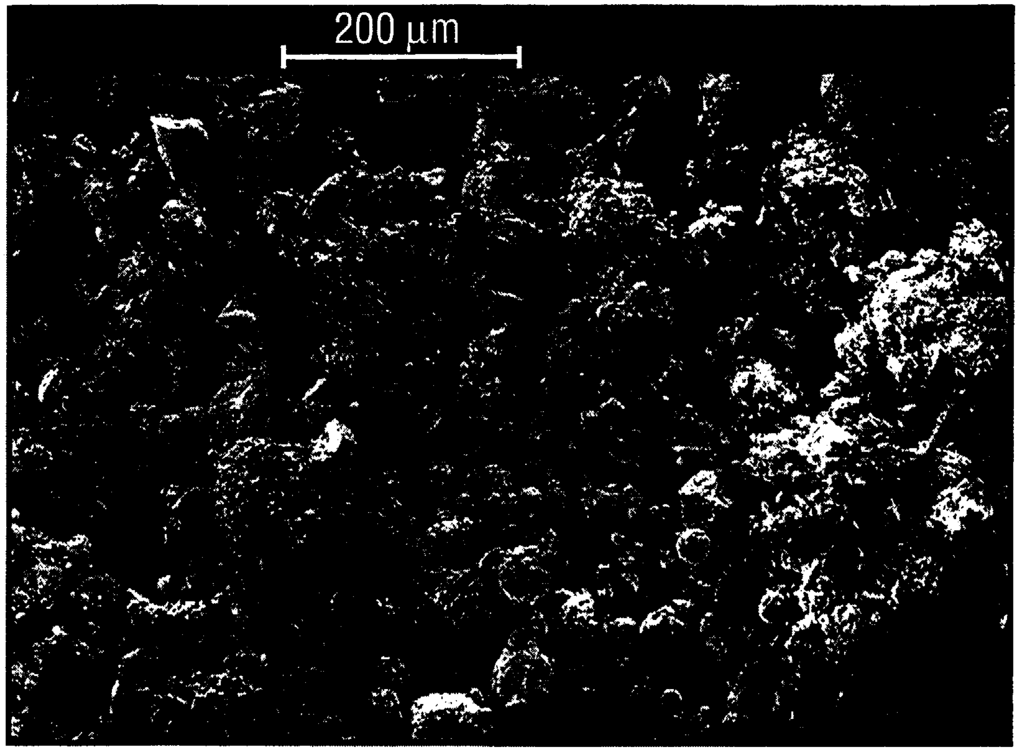


FIG. 2

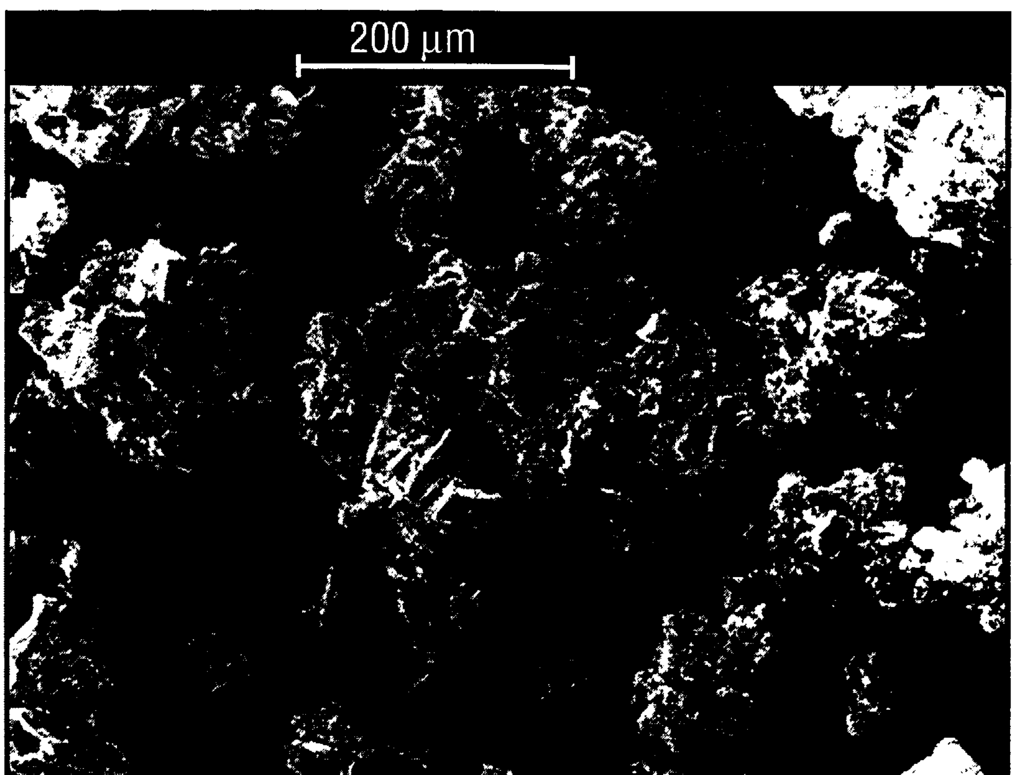


FIG. 3

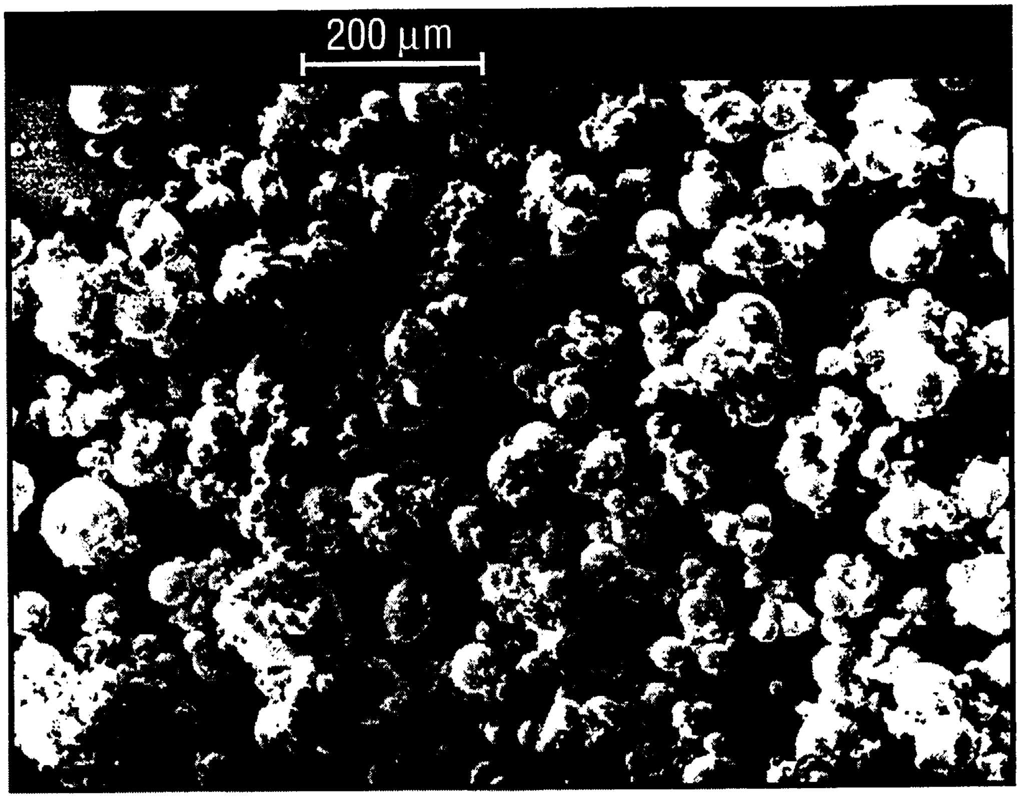
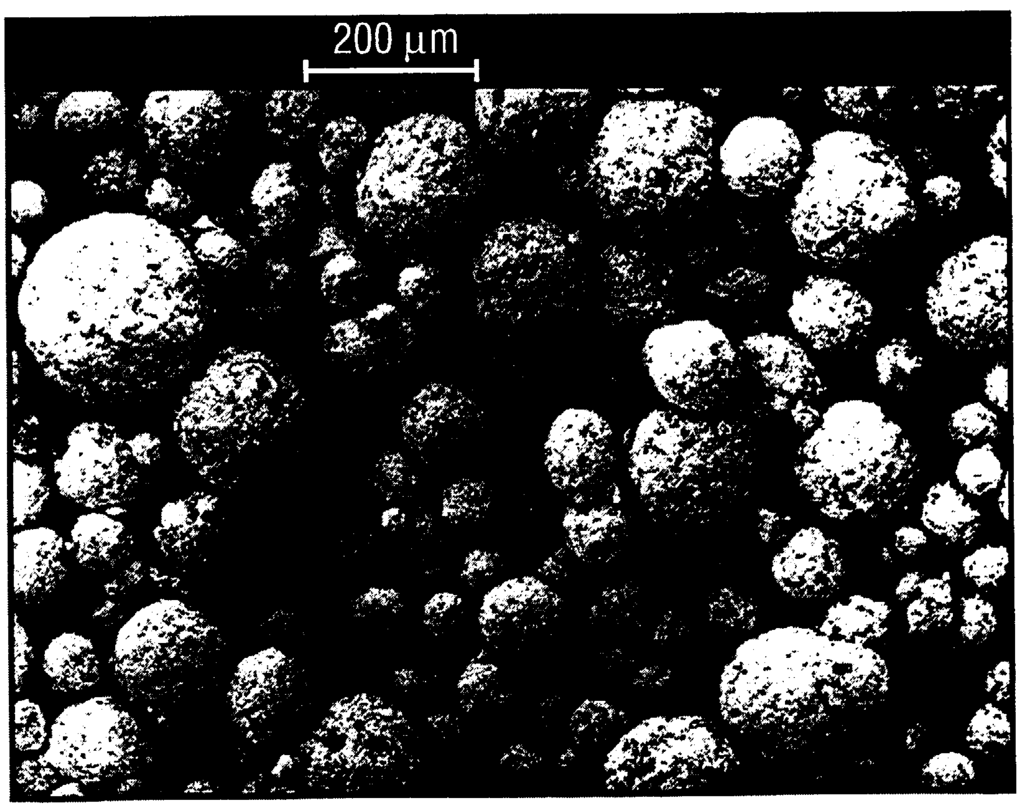


FIG. 4



METHOD OF SPRAY DRYING

This invention relates to a spray drying process which is particularly suitable for the spray drying of foodstuffs such as sugars. The invention also relates to the products of this spray drying process.

Spray drying processes are well known in the art. Generally, an aqueous dispersion of the material which is to be dried is sprayed into a hot, dry stream of gas passing through a drying chamber and the dried solids are collected. The spray drying process is used in the food industry for drying products such as milk, cream, instant coffee, cocoa, fruit and vegetable juices, extracts and flavourings. Other products such as drugs, detergents, soaps and cosmetics have also been dried in this manner.

Spray drying has also been used for the production of directly compressible forms of sugars such as lactose, sorbitol, dextrose and maltose. A directly compressible form of a material is useful since this physical form allows the material to be formed into tablets, without the use of binders or an intermediate granulation step. Certain sugars produced by a spray drying process are directly compressible. However, other sugars produced by conventional spray drying, such as sucrose, are not directly compressible. The requisite properties for a material to be directly compressible are not well understood.

One method of spray drying maltose is described in US patent 4,595,418. A process for producing powdery crystalline maltose by spray drying is described. The process is characterised by the preparation of the maltose solution to be spray dried and the low hygroscopicity of the product produced by this process.

However, it is difficult to use conventional spray drying techniques with certain materials, such as sucrose. When a sucrose solution is spray dried using conventional techniques, it rapidly forms an amorphous glass with water incorporated. The glassy sucrose will become stuck to the sides of the spray drying tower and any product collected will be sticky and hygroscopic. The hygroscopic sucrose products will be unsuitable for use in most commercial applications and are not directly compressible materials. Some other sugars, and also other materials including proteins and gums, may also form hygroscopic glasses in a similar way when spray dried. These hygroscopic glasses are not directly compressible.

Various attempts have been made to address the problems associated with spray drying sucrose. For example, GB-A-1240691 describes a spray drying process for a solution of sucrose, wherein a proportion of solid spray-dried sucrose is recycled and introduced into the spray drying chamber as seed particles. These seed particles act to produce a microcrystalline spray dried product. The sucrose solution has a solid content of at least 70% by weight and is typically saturated, or even super-saturated. However, the product of this process may be compressed into moulds and tablets only when it is still soft and hot.

GB-A-1386379 describes a method of spray drying a sucrose solution, wherein recycled solid product is introduced into the spray drying chamber. The spray drying chamber has a heated inlet at a temperature of at least 204°C, typically 218°C to 246°C, and the sucrose-containing solution has a solids content of from 60 to 90% by weight. It is alleged that this method substantially reduces the drying time in the spray drier.

US patent 3,615,723 describes a spray drying process wherein a screen is positioned to receive the spray. The spray forms a mat on the screen and hot air is drawn through the mat for the purposes of drying the material deposited on the screen. Some of the dried material may be recycled and introduced into the spray drying chamber in solid form. It is alleged that this process improves drying efficiency.

These processes are difficult to carry out in practice. Moreover, none of the processes is capable of producing spray dried sucrose which is directly compressible. Other technologies are used commercially to produce directly compressible sucrose, such as co-crystallisation (Domino DI-PAC (Registered Trade Mark), Tate & Lyle MICROTAL (Registered Trade Mark)), fluid bed agglomeration (Sudzucker COMPRISUGAR (Registered Trade Mark)) or wet granulation. Currently, there are no directly compressible sucrose products which are produced using spray drying technology.

It is an object of the present invention to provide a widely applicable spray drying process which is capable of producing stable food products which are free flowing, non-hygroscopic and directly compressible. It is a further object of this invention to provide a spray drying process which is capable of producing brown sugars with these same properties and which have a high molasses content. It is yet a further object of the present invention to provide a process which is capable of producing tabletted, coloured sugar products which do not suffer from undesirable mottling.

Accordingly, the present invention provides a spray-drying process comprising: providing a dispersion of a first material in a liquid solvent at a concentration of from 1 to 50% w/v; providing finely divided solid particles of a second material; spray drying the solution while continuously introducing the finely divided solid particles into the spray drier in an amount of from 10 to 99% by weight, based on the weight of the final product; whereby the process produces composite particles consisting of a spray-dried matrix of the first material bonded to particles of the second material.

Preferably, the first material comprises a sugar, a gum or a protein. More preferably, the first material comprises a sugar or sugar-based substance. Examples of suitable sugars or sugar-based substances include molasses, honey, invert, maltodextrin, glucose syrup, sucrose, glucose, fructose, lactose, sorbitol, dextrose, maltose, polyols and mixtures thereof. More preferably, the first material consists essentially of sucrose, invert, maltodextrin, lactose, molasses or mixtures thereof. Preferably, the solvent in which the first material is dispersed is water.

In one aspect of the present invention, there is provided a dispersion of the first material together with colourings and/or flavourings. Examples of suitable colourings and flavourings are Eurolake Sunset Yellow, Orange Flavour, Eurolake Blue and mint oil. The colourings and/or flavourings may be added to the solution of the first material in the presence of surfactants and/or emollients to improve their solubility.

Preferably, the second material comprises finely divided particles of a sugar. More preferably, the second material comprises sucrose, trehalose, fructose, maltodextrin, glucose syrup solids, or mixtures thereof. In one aspect of the present invention, the second material has been prepared by a spray drying process. In this aspect of the present invention, the second material may be recycled spray dried material.

Preferably, the second material consists essentially of finely divided crystalline particles.

Preferably, the mean particle size of the second material is from 1 to 450 μm. More preferably, the particle size is from 1 to 50 μm. Some preferred materials are sucrose in the form of ordinary icing sugar (mean particle size of about 25 μm) or finer icing sugar (mean particle size of about 10 μm), such as SILK (Registered Trade Mark of British Sugar PLC). Other suitable sugars include caster sugar (mean particle size of about 300 μm). The mean particle size may be determined by any suitable technique such as Laser scattering.

Preferably, the amount of second material is added in an amount ranging from 25% to 99%, more preferably 40% to 99%, more preferably 50% to 99%, and more preferably 70% to 99% by weight, based on the weight of the final product. Most preferably, the amount of second material is added in an amount ranging from above 80% to 99% by weight, based on the weight of the final product.

Preferably, the dispersion of the first material has a concentration in the range of 5% to 50% w/v. More preferably, the concentration of the dispersion of the first material is in the range of 5% to 20% w/v. These low concentrations of the dispersion gives the product desirable physical and chemical properties e.g. free flowing, low bulk density, improved colour intensity and improved dispersion.

Preferably, the inlet temperature of the air in the spray drier is in the range of 140 to 220°C. More preferably, the inlet temperature is in the range of 160 to 200°C.

In one aspect of the present invention, there is provided a spray-drying process, wherein: the first material is sucrose dissolved in water at a concentration of 5 to 50% w/v; the inlet temperature of the sucrose dissolved in water is about 190°C; the second material is a powdered crystalline sucrose having a mean size of 5 to 50μm, and the second material is added in an amount of from 80% to 90% based on the weight of the final product. In an alternative embodiment of this aspect of the present invention, the first material is sucrose dissolved in water which additionally comprises a colouring and/or flavouring dissolved in the same solution of water.

In another aspect of the present invention, there is provided a spray drying process wherein: the first material is molasses dissolved in water at a concentration of 5 to 50% w/v; the inlet temperature of the air is about 190°C; the second material is a powdered crystalline sucrose having a mean particle size of 5 to 50 μm; and the second material is added in an amount of from 80% to 95% by weight based on the weight of the final product.

The present invention also provides a free flowing particulate material obtainable by a process according to the present invention, wherein at least a fraction of the particles comprise a spray dried matrix of a first material bonded to particles of a second material.

The present invention also provides a particulate material comprising composite particles of a substantially amorphous matrix of a first material bonded to one or more particles of a second material, and further comprising spheroidal particles of the first material.

Preferably, the particles of the second material which are bonded to the first material are crystalline.

Preferably, the particulate materials according to the present invention are directly compressible and have low hygroscopicity. By directly compressible, it is meant that the particulate material compresses very readily with the minimum of force to produce a strong, robust compact. More preferably, the particulate materials are substantially non-hygroscopic.

In one aspect of the present invention, the particulate material consists essentially of sucrose.

In another aspect of the present invention, the particulate material is brown sugar. The brown sugar may have a molasses content of up to 20% w/w on a dry weight basis. Preferably, the molasses content is in the range of 5% to 15% w/w on a dry weight basis.
In a further aspect of the present invention, the particulate material is a coloured and/or flavoured sugar, gum or protein product. Preferably, the coloured and/or flavoured food

product consists essentially of a sugar. Preferably, the coloured and/or flavoured sugar, gum or protein product may be tabletted without significant amounts of mottling.

The present invention further provides a method of tabletting comprising direct compression of a particulate material according to the present invention. The present invention further provides tabletted products obtainable from this method of tabletting.

Specific embodiments of the present invention are now described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 shows a Scanning Electron Micrograph (SEM) of a product produced by the process of the present invention comprising spray dried invert (5% - dry solids) and icing sugar (95%).

Figure 2 shows a SEM of icing sugar particles produced by a conventional fluid bed agglomeration process.

Figure 3 shows a SEM of particles of trehalose produced by conventional spray drying.

Figure 4 shows a SEM of particles of maltose produced by a spray drying process described in US 4,595,418.

The process of the present invention is performed using the general procedure described below on a conventional spray drier having at least two inlet ports.

A vibratory feeder is placed at a first inlet port and the dry material flow rate calculated. When the first port is opened, the material is pulled by negative pressure into the spray drying chamber. This is performed as a solution of a second material is fed into a second inlet port and is spray dried. The spray dried product is taken and collected at the base of an adjoining cyclone.

A summary of the results obtained in Examples 1-20, together with all experimental parameters is shown in Table 1.

Table 1

Example	Details	Dry Feed Ingredient (2)	Wet Feed Ingredient (1)	Concn. of (1), %	Final Product (2) %	Final Product (1) %	Inlet Temperature, °C	Outlet Temperature, °C	% Moisture Content Karl Fischer
	100% Sucrose								
1		NCP	Sucrose	8	90	10	170	70	0.49
2		NCP	Sucrose	8	90	10	190	90	0.3
3		NCP	Sucrose	8	80	20	190	90	1.24
4		Caster	Sucrose	8	90	10	190	90	0.16
5		Granulated	Sucrose	8	90	10	190	75	0.38
	Experimental design								
6		NCP	Invert	8	80	20	190	70	0.6
7		NCP	Invert	8	90	10	190	70	0.42
8		NCP	Mixture	8	89.5	10.5	190	90	0.53
9		NCP	Mixture	40	89.5	10.5	190	85	0.25
10		NCP	Maltodextrin	8	94	6	190	85	0.62
11		NCP	Lactose	8	95	5	190	80	0.42
	Browns								
12		Caster	Molasses	8	92	8	190	90	0.22
13		Caster	Molasses	8	88	12	191	88	0.27
14		Granulated	Molasses	8	88	12	190	80	0.21
15		Granulated	Molasses	40	88	12	190	75	0.23
	Coloured and Flavoured								
16	50ppm Eurolake Sunset yellow	NCP	Sucrose	8	90	10	190	80	0.31
17	as16(i) + 0.2% orange flavour	NCP	Sucrose	8	90	10	190	80	0.23
18	as 16(i) + yellow and red food colours	NCP	Sucrose	8	90	10	190	80	0.23
19	300pm Eurolake blue	NCP	Sucrose	8	90	10	190	75	not measured
20	as 17(i) + 0.36 mint oil	NCP	Sucrose	8	90	10	190	75	not measured

NCP = 100% sucrose icing sugar (No Calcium Phosphate)

Examples 1-5 (100% Sucrose Products)

Example 1

An 8% sucrose solution ("wet feed ingredient") was spray dried as NCP icing sugar ("dry feed ingredient") was continuously introduced into the spray tower. (NCP icing sugar is a 100% sucrose product with a mean particle size of 20-25 μm, containing no calcium phosphate anticaking agent). The inlet temperature of air was about 170°C. The process was left to run for approximately 1 hour. When the spray drying chamber was inspected, there was no sucrose stuck to the exterior walls as is usually associated with sucrose spray drying. The material collected was more free flowing than icing sugar. The final product consisted of 90% dry feed ingredient and 10% wet feed ingredient, based on the amounts of dry solids in the starting material.

Example 2

Example 1 was repeated using conventional spray drying temperatures. The inlet temperature was about 190°C. Similar results were obtained as those from Example 1.

Example 3

Example 2 was repeated with the difference that the dry feed ingredient and wet feed ingredient were introduced into the spray drying tower such that the final product consisted of 80% dry feed ingredient and 20% wet feed ingredient, based on of the amounts of dry solids in the starting material.

Example 4

Example 2 was repeated with the difference that the dry feed ingredient was caster sugar. The product obtained displayed very different flow characteristics to caster sugar and was found to be more compressible than caster sugar.

Example 5

Example 2 was repeated with the difference that the dry feed ingredient was granulated sugar.

Examples 6-11

These Examples were performed in accordance with the process parameters shown in Table 1.

Example 6 produced excellent visible agglomerates and excellent flow characteristics. Some sticking on the exterior walls of the spray drier was observed.

Example 7 produced a good flowable powder with smaller agglomerates than Example 6. No processing problems were observed.

In Examples 8 and 9, "mixture" refers to a mixture of sucrose, invert, maltodextrin, and lactose. Example 9 used a 40% w/v wet feed solution. The product was finer than those Examples using an 8% w/v wet feed solution and had poorer flow characteristics.

Examples 12-15 (Brown Sugar Products)

These Examples were performed in accordance with the process parameters shown in Table 1.

Molasses was added to both caster and granulated sugars in order to determine how uniformly and effectively the spray dried material sticks to the sugar crystals.

Examples 12 and 13 both successfully produced a spray dried brown sugar product at 8% and 12% molasses on dry product. The product was more flowable than normal brown sugar. This is due to the lower moisture content and possibly the spray dried material rounding the edges of the caster crystals. Both samples were tacky when collected and needed to be broken up until dry to maintain their flow.

Examples 14 and 15 employed granulated sugar. Some separation of the spray dried material from the sugar crystals was observed.

Examples 16-20 (Coloured and Flavoured Products)

These Examples were performed in accordance with the process parameters shown in Table 1. All of Examples 16-20 tabletted successfully to produce tablets with little mottling.

Examples 16-18 produced products with solely orange colour and also with orange flavour. Both the colours and flavours were added to the solution of sucrose to be spray dried (wet feed ingredient).

In Example 17, addition of orange flavour produced an orange dextrose type flavour.

In Example 18, the colour of the yellow flavour was intensified by the addition of food colours.

Examples 19 and 20 produced strong deep blue coloured powders. In Example 20, mint oil was added to the sucrose solution, in addition to the blue colouring. Emulsifiers and/or surfactants may optionally be added to the solution to improve mixing.

The above Examples demonstrate some of the advantages of the present invention over the processes known in the prior art. All of the Examples produced free flowing, substantially non-hygroscopic products which were compressible and could be tabletted successfully to produce tablets.

A particular advantage illustrated in Examples 12 to 15 is the low moisture content of the brown sugar products. This produces brown sugar products which are more flowable than those products made by conventional methods. Brown sugar is currently produced by adding a volume of molasses onto a mixer containing the appropriate weight of sugar of suitable particle size (e.g. caster, granulated or medium sugar).

A further advantage illustrated in Examples 13 to 15 is that the process of the present invention enables the production of high molasses content sugars. Such sugars have a market in, for example, Christmas cake manufacture. Examples of typical brown sugar products are soft light brown (caster, 0.5% moisture, 2% molasses), soft dark brown (caster, 2.5% moisture, 8% molasses), and demerara sugar (medium sugar, 0.5% moisture, 1% molasses). Using conventional methods, the level of 8% molasses on the soft dark brown cannot be increased due to the viscosity of the molasses used. Any higher addition rates and the molasses will not stick to the sugar crystal. However, brown sugars produced according to Examples 13 to 15 of the present invention have a molasses content of 12%. In particular, Example 13, having 88% caster and 12% molasses, showed good flowability with no separation of the molasses from the caster being observed.

A particular advantage illustrated in Examples 16 to 20 is that little or no mottling was observed when the sugar products were tabletted. Traditionally, tabletted confectionary is very difficult to colour. In conventional processes, dry powders are added to the agglomerate before tabletting which results in a mottled and pastel coloured tablet. By contrast, in the present invention (Example 16 to 20), the colour is added to the wet feed solution prior to spray drying. Thus, the colour becomes an integral part of the agglomerate, and upon tabletting a stronger coloured tablet is produced without mottling.

Furthermore, the process of the present invention is cheaper than conventional processes in some cases.

With these advantages of the present invention in mind, it is envisaged that the process of the present invention can be applied advantageously to a produce a number of food products. These food products will have improved properties compared to products made by conventional methods. In some cases (e.g. honey), novel, powdered forms of a natural liquid food product will be available.

Suitable products include brown sugar, dry and icing fondant mixes, honey, invert, glucose syrup, sucrose, fructose, maltose, lactose, polyols, chocolate crumb, sweeteners, instant gums, instant proteins, beverage/dessert mixes. Such products will be available in a stable, free flowing, substantially non-hygroscopic, compressible, and optionally coloured form.

Without wishing to be bound by theory, it is believed that the inventive process produces novel products with the above-mentioned advantages by the way in which the solid ingredient in the spray drier coats the particles of spray dried material while they are still wet.

Scanning electron microscopy (SEM) shows that the final spray dried product consists mainly of crystalline particles of the solid ingredient bonded to the particles of spray dried ingredient in glassy form. Thus, the product differs fundamentally from the materials produced by seeding in the prior art (e.g. GB 1,240,691) which are essentially crystalline. The structure of the spray dried products typically produced by the present invention can be seen in Figure 1. There is a combination of mostly coral-like composite particles together with a few glassy spheroidal particles. It is believed that this unique structure gives the material its inherent direct compressibility.

By contrast, Figure 2 shows crystalline coral-like structures produced by a conventional fluid bed agglomeration process. No glassy spheroidal particles are present in this structure.

In Figure 3, there are the usual agglomerated glassy spheroidal particles produced by a conventional spray drying process.

In Figure 4, it can be seen that an alternative spray drying process, as described in US 4,595,418, produces a structure with glassy spheroidal particles coated with microcrystalline material.

Thus, it has been demonstrated that the process of the present invention produces a particulate material which has a unique structure, as compared to other spray drying or fluid bed agglomeration processes.

It will, of course, be understood that the present invention has been described by way of example, and that modifications of detail can be made within the scope of the invention.

Claims:

1. A spray-drying process comprising: providing a dispersion of a first material in a liquid solvent at a concentration of from 1 to 50% w/v; providing finely divided solid particles of a second material; spray drying the dispersion while continuously introducing the finely divided solid particles into the spray drier in an amount of from 10 to 99% by weight, based on the weight of the final product; whereby the process produces composite particles consisting of a spray-dried matrix of the first material bonded to particles of the second material.

2. A spray-drying process according to claim 1, wherein the first material comprises a sugar, a gum or a protein.

3. A spray-drying process according to claim 2, wherein the first material comprises a major fraction of a sugar.

4. A spray-drying process according to claim 3, wherein the first material consists essentially of sucrose, invert, maltodextrin, lactose, molasses or mixtures thereof.

5. A spray-drying process according to any preceding claim, wherein the dispersion of the first material additionally comprises colourings and/or flavourings.

6. A spray-drying process according to any preceding claim, wherein the dispersion of the first material has a concentration in the range of 5% to 50% w/v.

7. A spray-drying process according to any preceding claim, wherein the inlet temperature of the air as it enters the spray drier is in the range of 140°C to 220°C.

8. A spray-drying process according to any preceding claim, wherein the second material consists essentially of finely divided crystalline particles.

9. A spray-drying process according to any preceding claim, wherein the second material comprises a sugar.

10. A spray-drying process according to claim 9, wherein the second material consists essentially of finely divided crystalline sugar.

11. A spray-drying process according to any preceding claim, wherein the mean particle size of the second material is from 1 to 450μm.

12. A spray-drying process according to claim 11, wherein the mean particle size of the second material is from 5 to 50μm.

13. A spray-drying process according to any preceding claim, wherein the first and second materials are the same.

14. A spray-drying process according to any preceding claim, wherein the second material is added in an amount of from 50% to 99% by weight, based on the weight of the final product.

15. A spray-drying process according to any preceding claim, wherein: the first material is sucrose dissolved in water at a concentration of 5 to 50% w/v; the inlet temperature of the air is about 190°C; the second material is a powdered crystalline sucrose having a mean size of 5 to 50μm, and the second material is added in an amount of 80% to 90% based on the weight of the final product.

16. A spray-drying process according to claim 15 wherein the first material is sucrose dissolved in water which additionally comprises a colouring and/or flavouring dissolved in the same solution of water.

17. A spray-drying process according to any one of claims 1 to 14, wherein: the first material is molasses dissolved in water at a concentration of 5 to 50% w/v; the inlet temperature of the air is about 190°C; the second material is a powdered crystalline sucrose having a mean size of 5 to 50μm, and the second material is added in an amount of 80% to 90% based on the weight of the final product.

18. A free flowing particulate material obtainable by a process according to any preceding claim, wherein at least a fraction of the particles comprise a spray-dried matrix of a first material bonded to particles of a second material.

19. A free flowing particulate material comprising composite particles of a substantially amorphous matrix of a first material bonded to one or more particles of a second material, and further comprising spheroidal particles of the first material.

20. A particulate material according to claim 18 or 19, which is directly compressible.

21. A particulate material according to claim 18, 19 or 20, which is substantially non-hygroscopic.

22. A particulate material according to any one of claims 18 to 21 wherein the particles of the second material which are bonded to the first material are crystalline.

23. A particulate material according to any one of claims 18 to 22 consisting essentially of sucrose.

24. A particulate material according to any one of claims 18 to 22 which is brown sugar.

25. A particulate material according to any one of claims 18 to 22 which is a coloured and/or flavoured sugar, gum or protein.

26. A method of tabletting a particulate material according to any one of claims 18 to 25 comprising a direct compression step.

27. A tablet obtainable by the method of claim 26.

The Patent Office 17

INVESTOR IN PEOPLE

Application No:	GB 0017525.7	**Examiner:**	Dr Fatema Sardharwala
Claims searched:	1-27	**Date of search:**	19 January 2001

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.S): C6B (B8), B1B (BKA2)

Int Cl (Ed.7): A23L 3/46; B01D 1/18; C13F 1/02

Other: Online: EPODOC, WPI, JAPIO

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	EP 0749770 A1	(STORK) whole document, esp. line 54 of column 6 to line 51 of column 7	18, 19
X	US 4162926	(VELTMAN et al.) whole document, esp. Examples	18, 19
X	US 3956009	(LUNDQUIST et al.) whole document, esp. line 66 of column 1 to line 18 of column 2; lines 57-66 of column 2 and Examples	1-4, 6-11, 13, 14, 18, 19 22, 23

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
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&	Member of the same patent family		