1. Process for build-up granulation of a powdered material and/or the coating of granules in a fluidised bed with a binder contained in a granulating liquid, wherein the powdered material is fluidised, at least in parts of its bulk, in a fluidised bed apparatus by means of a gas current introduced from below through a perforated base and the granulating liquid is sprayed into fluidised zones of the material, wherein the granulating liquid is sprayed by means of flat jet nozzle(s) into fluidised zones of the material from above the fluidised bed, substantially in counter-current to the gas current which forms the fluidised bed.
Internationale Patentklassifikation: B01J 2/00, 2/16, A23K 1/00


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Titel: PROCESS AND DEVICE FOR PRODUCING GRANULATES FROM POWDERY MATERIAL

Bezeichnung: VERFAHREN UND VORRICHTUNG ZUM ERZEUGEN EINES GRANULATES AUS PULVERFÖRMIGEM MATERIAL

Abstract

A process is disclosed for constructively granulating a powdery material in a fluidized bed equipment. The granulating liquid containing a binder is sprayed downwards on the fluidized bed of material to be granulated in countercurrent to the fluidized bed-generating gas by means of two-component fan jet nozzles. With charge quantities on the order of about 1000 kg, very uniform and dust-free granulates can thus be obtained.

Zusammenfassung

Boehringer Ingelheim Vetmedica GmbH,
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Process and apparatus for producing granules from powdered material
Specification

The invention relates to a process for build-up granulation of a powdered material and/or the coating of granules in a fluidised bed with a binder contained in a granulating liquid, wherein the material is fluidised in a fluidised bed apparatus by means of a gas current blown in from below through a perforated base, at least in parts of the bulk of the material, and the granulating liquid is sprayed into fluidised zones of the material. The invention further relates to an apparatus for carrying out such a process.

Pharmaceutical compositions, feed additives and other products are frequently obtained, at the end of the manufacturing process itself, as a powder or powder mixture of a specific particle size distribution. Even a product which is chemically or in some other way specifically suited for a particular use may occasionally fail to develop its full activity under practical conditions or may be restricted in its handling qualities if it is not presented in a suitable external form. The solution to problems of inadequate chemical resistance or hazardous handling of a powdered material on the grounds of dust formation may in certain cases consist in granulating the powdered material and optionally also coating the granulate particles with a protective substance which has no inherent drawbacks.

EP-A-165 577 describes, in the case of a feed additive containing zinc bacitracin, how granulation can substantially improve the chemical stability of a feed additive of this kind in a feed mixture. The preferred method of granulation proposed according to EP-A-165 577 is the use of a fluidised bed granulator in which the powdered starting material is placed and, after a fluidised bed has been produced by blowing a gas current
through a perforated base on which the starting substance rests, a granulating liquid is sprayed into the fluidised bed by means of spray nozzles, this liquid containing 1-10% by weight of a suitable binder, in solution or suspension, which will bring about increased agglomeration of the powdered material to form granules and finally coats these granules with a protective layer.

Whereas, in the subject matter of EP-A-165 577, the essential objective was to improve the chemical stability of a feed additive containing zinc bacitracin in a feed mixture, EP-A-302 462 describes an improved granulating process for feed additives in general, wherein a slotted gyro dynamic filter granulating apparatus is used for the granulation. An apparatus of this kind is described in DE-A-29 32 803, to which reference is also made in EP-A-302 462, and, in a somewhat different embodiment, in the later EP-A-331 111. This is a fluidised bed apparatus in which, underneath the perforated base, in the wind chamber, there is a rotor which only allows the fluidising gas to be blown in in certain radially extending zones which advance slowly as a result of the rotation of the rotor, so that fluidised bed areas can only form in certain sector-shaped areas and migrate slowly through the bulk of the material.

Whereas, in the apparatus according to DE-A-29 32 803, there is only a single spray nozzle arranged centrally above the perforated base to spray the coating or granulating liquid into the fluidised bed from below, in the apparatus according to EP-A-331 111 there are radial arms provided above the perforated base, having upwardly directed spray nozzles which rotate synchronously with the slotted gyro rotor, so that they migrate together with the fluidised bed zones and spray the granulating
liquid into them from below.

EP-A-302 462 describes, again taking the example of a feed additive containing zinc bacitracin, how the granules produced in the slotted gyro fluidised bed apparatus not only have even better chemical stability but also a very narrow and even particle size distribution of the granules, which contain hardly any oversized particles and are virtually free from dust. The dust produced in an abrasion test, moreover, contains hardly any of the active substance, zinc bacitracin.

As is clear from the embodiments by way of example in EP-A-302 462, batches of starting material of the order of 3 kg were processed. Feed additives are needed in tonnes. To enable economical production it is desirable to produce granules in amounts ranging from several hundred kilograms up to one tonne.

The aim of the invention is to provide a process for build-up granulation of powdered material and a suitable apparatus which will produce, more particularly on an industrial scale, uniform and substantially dust-free granules which correspond in their properties to the results given in EP-A-302 462.

It is well known to those skilled in the art that the flow characteristics of substances depend significantly on the external conditions and also on the apparatus used. This also applies in each case to granulating processes using fluidised bed apparatus. The experiments were carried out with a fluidised bed apparatus approximately 2 m in diameter, the batches of powdered material weighing about 750 kg. However, it is assumed that the results achieved can also be transferred analogously to apparatus of different orders.
of magnitude. The experiments were also carried out in this instance with a feed additive containing zinc bacitracin, the composition and other conditions of which can be found in EP-A-165 577 and EP-A-302 462, to which reference is expressly made. Even though the experiments carried out were intended primarily for granulating a feed additive containing zinc bacitracin on an industrial scale, with specific given properties, the present invention is not restricted to a feed additive of this kind but is generally suitable for the granulation of powdered material, the starting materials and granulating liquids being as described in EP-A-302 462, to which reference is expressly made for this reason.

Other substances of particular interest are cimaterol, sputolysine, bisolvone, virginiamycin, tylosin, spiramycin, nosiheptide, penicillins, chlorotetracyclines, oxytetracyclines, tetracyclines, erythromycins, furazolidone, nitrofuran, thrimethoprim, sulphonamides, dimetridazole, neomycin base and all vitamins which are pharmaceutically accepted or allowed under food regulations, such as vitamin A, A₁, A₂, B₁, B₂, B₄, B₆, B₁₂, C (ascorbic acid), ascorbylpalmitate and other pharmacologically acceptable derivatives of ascorbic acid, D, D₁, D₂, D₃, D₄, E, H, K, K₁, K₂, P and Q - or active substances such as avoparcin, flavopholipol, monensin, monensin-sodium, salinomycin, carbadox, nitrovin and olaquindox.

The objective of improving a process of the type described hereinbefore is achieved according to the invention in that the granulating liquid is sprayed into fluidised zones of the material by means of flat jet nozzles from above the fluidised bed, substantially in counter-current to the gas current which forms the fluidised bed. A fluidised bed apparatus which is
suitable for performing the process according to the invention is accordingly characterised in that the spray nozzles used are flat jet nozzles which are arranged in an upper part of the container which, when the apparatus is operated as intended, is above the fluidised bed formed, and these flat jet nozzles are directed so that their direction of spraying is substantially downwards towards the fluidised bed.

On an industrial scale, using a larger apparatus, it has been found that spraying the granulating liquid in from below, i.e. from the perforated base upwards into the fluidised material zones, is obviously not the optimum method. Particularly if a fluidised bed has been formed only in certain areas, as with the slotted gyro method, the fluidising gas appears to carry up the granulating liquid which has been blown in in parallel, so that it does not come into optimum contact with the material which is to be agglomerated. Therefore, it has proved more effective to spray the granulating liquid in from above, so to speak in counter-current to the fluidising gas, into the fluidised areas. The best results were obtained with flat jet nozzles which, in the case of a container of circular cross-section, are preferably aligned so that the elongate surface covered by the jet runs with its long main axis substantially in the direction of a radius of the apparatus.

The most effective nozzles have proved to be two-component flat jet nozzles with external mixing, wherein, after leaving the nozzle, the liquid which is to be sprayed is atomised by an auxiliary gas current and brought into the desired jet form. The reasons for the advantageous effect of these nozzles has not hitherto been explained in detail but it is presumed that, in spite of the auxiliary pressure of the atomising gas, a soft, well distributed jet is produced
with a droplets dynamic which produces a particularly even agglomeration effect in conjunction with the fluidised powdered material.

It has also been found that, if a suitable number of flat jet nozzles are distributed over the fluidised bed, it is no longer necessary in general to use the rotating slotted gyro, i.e. to produce limited fluidised bed areas which rotate slowly through the apparatus. Good results are achieved almost to the same extent with a perforated base which is fully acted upon by fluidising gas. Moreover, it is not absolutely essential to have the flat jet nozzles migrating over the fluidised bed, although this is a preferred embodiment of the process according to the invention. Whether this measure is necessary depends on the one hand on the number and distribution of the nozzles and on the other hand on the dynamics of the fluidised bed. It has been found that, even with stationary flat jet nozzles which cover only about 12% of the surface of the fluidised bed, uniform granules can be achieved. The duration of spraying and the recirculating effect in the fluidised bed presumably contribute to the results of the process.

The choice of the number of nozzles is not free in as much as a limited amount of granulating liquid has to be sprayed into a batch of material, and the spraying must extend over a certain length of time in order to achieve slow agglomeration and granulation, whilst the fluidising gas must also be in a position to remove the solvent or suspension agent from the granulating liquid. Since on the other hand the nozzles used must have a specific amount travelling through them, it is not possible to spray the entire surface of the fluidised bed. Preferably, according to the invention, at any one time, only about 10-25% of the surface of the fluidised bed is covered by the spray jets of the nozzles. This
fact may make it necessary to allow the nozzles to migrate over the fluidised bed or rotate, to achieve better spray distribution.

Generally, 2.5-30% by weight, preferably 5-20% by weight, based on the finished granules, of binder will be introduced into the product, the granulating liquid being a 1-30%, preferably 2-10% and more especially 5-7% solution or suspension of the binder.

In one particular alternative embodiment of the process it is possible first to spray the pure active substance on to the carrier material and to coat it with the coating material in a second step. In this alternative embodiment the proportion by weight of active substance is between 0.01 and 30% by weight, based on the finished granules, depending on the pharmacological activity of the active substance. The active substance may be sprayed on in the form of a solution, for example a 0.01 to 30% by weight solution, or in the case of less soluble active substances, in the form of a suspension. When adjusting the concentration of the solution containing the active substance, obviously the nature of the solvent and the solubility of the active substance in the solvent must be borne in mind.

This alternative embodiment is of particular interest with highly active substances such as clenbuterol or spuolysine. Examples of carriers include inorganic carriers such as calcium carbonate or dicalcium phosphate, and examples of organic carriers include sugars such as lactose, lactose-corn starch or saccharose, but also sorbitol.

In the experiments carried out with the feed additive, about 5% of methyl cellulose, based on the finished granules, were used, the granulating liquid being a 5%
aqueous solution of the methyl cellulose. Other coating materials are possible, as disclosed for example in EP-A 165 577 and 302 462, to which reference is made.

Of particular interest, apart from methyl cellulose, are hydroxypropyl cellulose, hydroxypropylmethyl cellulose, polyethyleneglycol and water soluble starch.

With a binder content of 5% and 5% solution, the total amount of granulating liquid in litres corresponds substantially to the weight in kg of the powdered material specified. In order to achieve good results, this quantity of liquid was preferably sprayed into the fluidised bed over a period of 3 to 4 hours. The quantity of liquid per unit of time need not necessarily be kept constant throughout the entire spraying time. Indeed, rather more liquid may be sprayed at the beginning of the process and rather less towards the end. With a quantity of about 1 litre of granulating liquid per kg of powdered material and a spraying time of just 3.5 hours, an average spray quantity per unit time of only about 5 ml/kg of starting material per minute is obtained. It follows from this that the spray density on the top of the fluidised bed cannot be maintained at a very high level.

Two-substance flat jet nozzles suitable for the process according to the invention are sold, for example, by the company Spraying Systems. In tests, one nozzle made by this company, bearing the name SUE 45 and having a spray angle of 45°C proved suitable, *inter alia*. According to the specification of the manufacturer this nozzle has a liquid throughput of 4.7 l/min for operation with water at a fluid pressure of 1.5 bar and an air pressure of about 6.0 bar. At a liquid inlet pressure of 0.7 bar and an air pressure of 5-6 bar, the water throughput falls to 3.2 l/min. In the case of higher viscosity
liquids, such as the granulating liquids involved here, the liquid throughput of the nozzles is substantially less. The viscosity of the liquid may also be affected by its temperature. It has been found that the nozzles operate satisfactorily with the granulating liquid if the viscosity of the liquid is adjusted so that the nozzles have a throughput which is 0.1-0.3 times, preferably 0.12-0.18 times the nominal throughput with water. Preferably, the nozzles are operated at a pressure of atomising gas of between 2 and 6 bar. The fluid pressure is determined by the desired throughput of the nozzle. It may vary between 0.7 and 3 bar and is preferably between 1.2 and 1.8 bar. With a 5% methyl cellulose solution having a temperature of 30°C, the throughput with the above-mentioned nozzle type is about 0.6-1.4 l/min.

Depending on the spacing of such a nozzle from the surface which is to be sprayed, in this case the theoretical surface of the fluidised bed, the nozzle sprays an area of 150-750 cm², a spraying area of between 500 and 700 cm² being preferred, for which the average spacing of the nozzle from the surface to be sprayed is about 70 cm. The preferred spray density, based on the area actually sprayed, is preferably between 5 and 15 l/m².min.

The spray power of the individual nozzles and the quantity of granulating liquid to be sprayed per unit of time will give the number of nozzles required, once a specific nozzle type has been chosen, whilst the vertical arrangement, in particular, will produce the preferred minimum area to be sprayed. In the case of an apparatus of circular cross-section the nozzles are preferably arranged so that their spray areas extend radially and are uniformly spaced over the periphery thereof. If the length of the spray area of a nozzle is
more or less sufficient to cover the radius of a container, a ring of nozzles is preferably used, the nozzles being spaced from the centre point roughly half the length of the radius. In the case of a container with a diameter of 2 m, for example, good results were achieved with six nozzles arranged in this way and distributed over the circumference. For larger container diameters, a larger number of nozzles may be required, and these nozzles, if they do not cover almost the entire length of the radius of a container, should be arranged alternately at different spacings from the centre of the container in such a way that the most uniform spray distribution possible is achieved over the fluidised bed. Based on the total surface area of the fluidised bed a spray density of between 1 and 3 l/m².min, preferably between 1.2 and 1.5 l/m².min has proved suitable.

To produce the fluidised bed, quantities of gas ranging from 10-60 m³/min.m² of perforated base have proved suitable. The process according to the invention has therefore proved particularly economical in this respect. Unlike many known methods, the process according to the invention is not particularly affected by whether the current of fluidising gas through the perforated base is generated by overpressure below the perforated base or by under-pressure above the perforated base. Therefore, for generating the gas current, either a pressure-generating blower may be provided in the gas chamber below the perforated base or a suction blower may be provided in the product space above the fluidised bed.

For processing the feed additive used in the experiments, good results were obtained by operating with a specific perforated base load of 200-300 kg of powdered starting material per m² of perforated base. In
this particular instance, the fluidising gas used was nitrogen, which was at a temperature in the range from 90 to 110°C, preferably 100°C, on entering the granulating apparatus. The powdered material was heated to a temperature above 70°C, preferably 80°C, before the start of spraying. The following is a description of the basic construction of an embodiment of a fluidised bed apparatus according to the invention, referring to the accompanying drawings, wherein:

Fig. 1 is a diagrammatic vertical section through the important components of the apparatus and

Fig. 2 is a diagrammatic horizontal section through the apparatus, showing the distribution of the spray nozzles.

The fluidised bed granulator shown in diagrammatic cross-section in Fig. 1 comprises a product container 2 for the powdered material which is to be granulated, sealed off at its lower end by a fine perforated base 4, the degree of fineness of which is such that the powdered material which it is intended to support cannot pass through the perforated base in any substantial amount. Above the product container 2 and on an upward continuation thereof is provided an upper section 6 of the granulator. This upper section has a cylindrical outer casing 8 which is sealed off at the top by an upper wall 10. Below the product container 2 or the perforated base 4 thereof is a wind chamber 12 provided with gas inlet ports 14 which are connected to a blower (not shown). In the upper section 6 of the apparatus is a gas outlet port 16 through which the fluidising gas can leave the apparatus again. The broken line 18 in the top part of the apparatus diagrammatically indicates a dust filter which is intended to catch any dust particles from the powdered material carried by the
fluidising gas and either return these particles to the product container from above or collect them for further treatment, depending on the particular design.

Along the central axis of the apparatus and in the lower part thereof is provided a shaft 22 which is rotatably driven by a motor 20, this shaft passing through the perforated base from below and being provided, above the perforated base, with spray arms 24 each of which is provided at its end with a two-substance flat jet nozzle 26. Figure 2 shows that the shaft 22 is fitted with six spray arms arranged at equal angular spacings around the circumference of the apparatus, these spray arms together carrying six spray nozzles which are positioned substantially halfway along the radial spacing of the shaft 22 from the outer wall of the apparatus. The shaft 22 and the spray arms 24 are of hollow construction in order to be able to supply spray liquid to the nozzles 26 by means of supply means (not shown).

In the drawings, the product container 2 is not provided with a charging port for the powdered material; rather, the whole thing including the perforated base 4 can be moved laterally out of the fluidised bed apparatus in order to load and unload it, as indicated by the break 28 in the outer wall of the apparatus. The apparatus is intended for batch operation.

In the embodiment of the fluidised bed apparatus used for experimental purposes, the diameter of the product container and of the upper section was about 2 m, which, after deduction of the cross-section of the shaft 22 above the perforated base, gave a perforated base area of about 3.1 m². The spray nozzles used were two-component flat jet nozzles made by the firm Spraying Systems, of the type SUE 45. The nozzles were arranged about 145 cm above the perforated base 4. With a
fluidised bed height above the perforated base of about 75 cm, this gave a spacing of the nozzles from the fluidised bed of about 70 cm. The nozzles were operated under a gas pressure of about 4 bar. Under a fluid pressure of about 1.5 bar, a nozzle of this kind has a water throughput of about 4.7 l/min. When spraying a 5% aqueous methyl cellulose solution the throughput per nozzle is about 0.6-1.4 l/min, depending on the fluid pressure.

The drive for the shaft 22 was designed to be regulatable for speeds between 0.2 and 10 rpm.

The fluidised bed apparatus described above was used to granulate a powdered feed additive containing zinc bacitracin. A typical embodiment by way of example is given hereinafter:
Embodyment by way of Example

712.5 kg of a powdered feed additive containing zinc bacitracin were placed in the product container of the fluidised bed apparatus described above. The powder had a particle size distribution of 1-40 μm, the majority of the particle size distribution being in the range from 10-15 μm. In addition to 5-30% by weight of zinc bacitracin a powder of this kind also contains fermentation residues from the manufacturing process and calcium carbonate.

Nitrogen was used as the fluidising gas. At the start of the process, the powdered starting material was heated to 80°C for about 20 minutes by means of nitrogen gas at a temperature of 95°C.

For the spraying of the granulating liquid which takes place next, six flat jet nozzles were used, with their jets directed downwards on to the fluidised bed, these nozzles rotating at a speed of 6 rpm through the apparatus. A total of 750 l of a 5% methyl cellulose solution at a temperature of 30°C was sprayed on.

In order to produce the fluidised bed, nitrogen gas at approximately 100°C was blown into the apparatus in increasing amounts, initially at a range of 40 m³/min and, towards the end, at a rate of 187 m³/min. The spray rate of the granulating liquid was 4 l/min at the beginning and was increased by 1 l/min after 20 litres had been sprayed in, and finally to 8 l/min. As the spray rate was increased the quantity of fluidising gas was also increased by 10 m³/min up to 90 m³/min. After 120 litres of granulating liquid had been sprayed on, the spray rate was reduced to 4 l/min and kept constant. The total spraying time was about 3 hours.
After spraying had finished and the nozzles had been washed with a certain quantity of water, the granules produced were dried. The product temperature and the spent air moisture of the dry air were monitored.

The particle size distribution of the granules was very even, the proportion of particles between 180 and 710 μm in the granules was more than 80%, which satisfied the specifications. More than 98% of the granules fell within the range of 125 to 1000 μm. The proportion of very fine particles measuring less than 45 μm was 0.01%, which was again within the specification.

Therefore, granules which are totally satisfactory can be produced using the method according to the invention and the fluidised bed apparatus according to the invention.

Analogously to the Example described, tests were also carried out on an industrial scale with calcium carbonate and lactose as carriers and further experiments were carried out using soluble starch as the coating material.
The claims defining the invention are as follows:

1. Process for build-up granulation of a powdered material and/or the coating of granules in a fluidised bed with a binder contained in a granulating liquid, wherein the powdered material is fluidised, at least in parts of its bulk, in a fluidised bed apparatus by means of a gas current introduced from below through a perforated base and the granulating liquid is sprayed into fluidised zones of the material, wherein the granulating liquid is sprayed by means of flat jet nozzle(s) into fluidised zones of the material from above the fluidised bed, substantially in counter-current to the gas current which forms the fluidised bed.

2. Process according to claim 1, wherein two-component overpressure flat jet nozzles are used for external mixing, which are operated at a pressure of atomising gas of 2 to 6 bar.

3. Process according to claim 1 or 2, wherein a plurality of flat jet nozzles are used which are distributed as evenly as possible over the surface of the fluidised bed.

4. Process according to at least one of claims 1 to 3, wherein the bulk of the material is fluidised only in parts and these parts and, together with them, the flat jet nozzles are allowed to migrate through the fluidised bed apparatus.

5. Process according to at least one of claims 1 to 3, wherein substantially all the bulk material is fluidised in the fluidised bed and the flat jet nozzles are allowed to migrate over the fluidised bed.
6. Process according to at least one of claims 1 to 5 for processing a feed additive containing zinc bacitracin, wherein, in the fluidised bed apparatus, a specific bulk of 200-300 kg of starting material is processed per m² of perforated base, and the granulating liquid used is a 4 to 6% by weight aqueous solution of methyl cellulose.

7. Fluidised bed apparatus having a container, a perforated base arranged in the container for carrying powdered material, a device arranged underneath the perforated base for introducing fluidising gas through the perforated base into the material and at least one spray nozzle arranged above the perforated base for spraying granulating liquid, as well as means for conveying the granulating liquid to the or each spray nozzle, wherein the or each spray nozzle is a flat jet nozzle which is arranged in an upper part of the container, which is located above the fluidised bed formed when the apparatus is operated as intended, and which is arranged so that its direction of spraying is substantially downwards towards the fluidised bed.

8. Fluidised bed apparatus according to claim 7, wherein the or each spray nozzle is a two-component overpressure flat jet nozzle with external mixing.

9. Fluidised bed apparatus according to claim 8, wherein the or each flat jet nozzle is designed for a pressure of atomising gas of 2 to 7 bar and for a pressure of 0.2 to 3.0 bar in the granulating liquid which is to be atomised.

10. Fluidised bed apparatus according to claim 8 or 9, wherein the or each flat jet nozzle is designed for a liquid throughput, based on water, of 1.5 to 5.0 l/min.
11. Fluidised bed apparatus according to at least one of claims 7 to 10, with a container of substantially circular cross-section, wherein the or each flat jet nozzle is arranged with its spraying area directed substantially radially, the spraying area, based on the vertical central axis of the container, extending only on one side of said axis substantially along a radius, and the spray nozzle being rotatable about the central axis of the container.

12. Fluidised bed apparatus according to claim 11, wherein a plurality of flat jet nozzles (26) are uniformly distributed over the circumference of the container (2, 6).

13. Fluidised bed apparatus according to claim 12, wherein said plurality of flat jet nozzles comprises 6-12 flat jet nozzles.

14. Fluidised bed apparatus according to claim 11, 12 or 13, wherein the flat jet nozzles are mounted by means of arms on a central rotation shaft.

15. Fluidised bed apparatus according to claim 14, wherein the means for supplying the granulating liquid to the flat jet nozzles comprises the hollow rotation shaft and the arms.

16. Fluidised bed apparatus according to at least one of claims 11 to 15, wherein, under the perforated base, there is a rotatable plate having substantially radial air passage slots which are rotatable in registry with the flat jet nozzles.

17. Fluidised bed apparatus according to at least one of claims 7 to 10 with a container of substantially circular cross-section, wherein a plurality of flat jet
nozzles are arranged with their spray areas substantially radially aligned at substantially equal spacings over the circumference of the container.

18. Fluidised bed apparatus according to claim 17, wherein 6 to 12 flat jet nozzles are provided.

19. Fluidised bed apparatus according to at least one of claims 12 to 18, wherein the area of the fluidised bed covered by the flat jet nozzles at any time is about 10 to 25% of the total area of the fluidised bed.

20. Fluidised bed apparatus according to at least one of claims 12 to 19, wherein the surface of the fluidised bed covered by a flat jet nozzle is 150 to 750 cm², preferably 600-700 cm².

21. Use of a fluidised bed apparatus according to one of claims 7 to 20, such use comprising effecting build-up granulation of a powdered material and/or the coating of granules with a binder contained in a granulating liquid.

22. Use of a fluidised bed apparatus according to one of claims 7 to 20, such use comprising effecting build-up granulation of a powdered feed additive containing zinc bacitracin.

23. Process for build-up granulation of a powdered material and/or the coating of granules in a fluidised bed with a binder contained in a granulating liquid, substantially as hereinbefore described with reference to the accompanying drawings.

24. Fluidised bed apparatus substantially as hereinbefore described with reference to the accompanying drawings.

DATED this 5th day of September, 1996.

BOEHRINGER INGELHEIM VETMEDICA GMBH
By their Patent Attorneys:
CALLINAN LAWRIE
### A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl.5  B01J 2/00;  B01J 2/16;  A23K 1/00

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)

Int.Cl.5  B01J;  A23K;  B05B

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)

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category*</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<tbody>
<tr>
<td>Y A</td>
<td>US, A, 3 382 093 (NACK) 7 May 1968 see column 8, line 36- line 58; claim 1; figure; examples 1-15</td>
<td>1, 3, 7-9 4, 5, 10, 11</td>
</tr>
<tr>
<td>Y</td>
<td>EP, A, 0 195 763 (LEJUS MEDICAL AKTIEBOLAG) 24 September 1986; see column 1, line 11- line 39; figure 2</td>
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<td>A</td>
<td>EP, A, 0 302 462 (BOEHRINGER INGELHEIM VATMEDICA) 8 February 1989; see page 3, line 31- line 35; examples 1, 2</td>
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<td>A</td>
<td>EP, A, 0 407 325 (A/ NIRO ATOMIZER) 9 January 1991; see claims 1-11; figure 3- 5, 7, 11, 12, 14</td>
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<tr>
<td>A</td>
<td>EP, A, 0 330 207 (WARNER-LAMBERT COMPANY) 30 August 1989; see claims 1-9; figure 2</td>
<td>1, 3-5, 7</td>
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Further documents are listed in the continuation of Box C. 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 document but published on or after the international filing date
- "L" document which may throw doubt on priority claim(s) or is cited to establish the publication date of another document or other special reason as specified
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Date of the actual completion of the international search: 8 September 1993 (08.09.93)
Date of mailing of the international search report: 29 September 1993 (29.09.93)

Name and mailing address of the ISA/European Patent Office

Authorized officer

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INTERNATIONALER RECHERCHENBERICHT
Internationales Aktenzeichen: PCT/EP 93/01328

I. KLASSIFIKATION DES ANMELDUNGSGEGENSTANDS (bei mehreren Klassefikationsymbolen sind alle anzugeben)*
Nach der internationalen Patentklassifikation (IPC) oder nach der nationalen Klassifikation und der IPC
Int.Kl. 5 B01J2/00; B01J2/16; A23K1/00

II. RECHERCHIERTE SACHGEBIETE

<table>
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<th>Klassefikationsystem</th>
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Recherchierte nicht zum Mindestprüfstoff gehörige Veröffentlichungen, soweit diese unter die recherchierten Sachgebiete fallen 8

III. EINSCHLAGIGE VERÖFFENTLICHUNGEN *

<table>
<thead>
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<th>Art.</th>
<th>Kennzeichnung der Veröffentlichung 11, soweit erforderlich unter Angabe der maßgeblichen Teile 12</th>
<th>Betr. Anspruch Nr. 13</th>
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<td>Y</td>
<td>US,A,3 382 093 (NACK) 7. Mai 1968</td>
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<td>A</td>
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<td>EP,A,0 195 763 (LEJUS MEDICAL AKTIEBOLAG) 24. September 1986</td>
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<td>siehe Seite 3, Zeile 31 - Zeile 35; Beispiele 1,2</td>
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15. BESCHREIBUNG
Datum des Abschlusses der internationalen Recherche
08. SEPTEMBER 1993
Abendsdaten des internationalen Recherchenerichts
29. 09. 93
Internationale Recherchenbehörde
EUROPAISCHES PATENTAMT
Unterschrift des bevollmächtigten Beispielsprechers
CUBAS ALCARAZ J.L.

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