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(54) **Titre : PROCÉDE ET DISPOSITIF DE PRODUCTION D'UN COMPRIME**
(54) **Title: PROCESS AND APPARATUS FOR PRODUCING A PELLET**

(57) **Abrégé/Abstract:**

The invention concerns a method and a device for producing a tablet which is preferably provided for subsequent material-determining analysis, wherein: a material flow is melted; the molten material flow is transformed and cooled to form a vitreous material; ground; and at least some of the ground material flow is compressed to form a compressed tablet.

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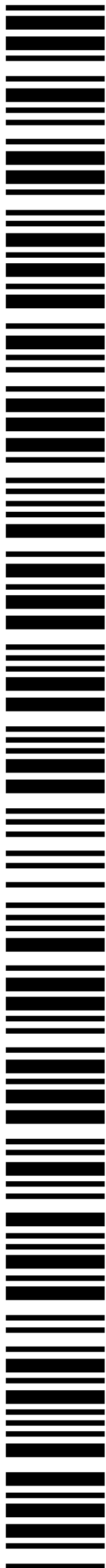
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(54) Title: METHOD AND DEVICE FOR PRODUCING A TABLET

(54) Bezeichnung : VERFAHREN SOWIE EINE VORRICHTUNG ZUR HERSTELLUNG EINER TABLETTE

(57) Abstract: The invention concerns a method and a device for producing a tablet which is preferably provided for subsequent material-determining analysis, wherein: a material flow is melted; the molten material flow is transformed and cooled to form a vitreous material; ground; and at least some of the ground material flow is compressed to form a compressed tablet.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren und eine Vorrichtung zur Herstellung einer Tablette, die vorzugsweise für eine spätere Analyse zur Stoffbestimmung vorgesehen ist, wobei ein Stoffstrom geschmolzen wird, der geschmolzene Stoffstrom zu einem glasigen Material ungeformt abgekühlt und vermahlen wird, und wenigstens ein Teil des vermahlenden Stoffstromes zu einer Presstablette gepresst wird.



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Process and apparatus for producing a pellet

The invention relates to a process and an apparatus for producing a pellet preferably intended for subsequent analysis to identify substances.

5 Producing pellets in the form of pressed pellets by grinding the pellet material and further processing said material by employment of pressure and/or binding agent to afford the pellet is prior art. A process of this type requires that, prior to grinding, the pellet material already has a consistency suitable for the finished pellet.

10 Producing pellets from a melt is also prior art. This comprises melting the pellet material, pouring the melt into a pellet mould and cooling it therein. However, such cooling with simultaneous shaping entails complexity both in terms of process engineering and in terms of apparatus. For instance, cooling of a fused pellet must be performed under precisely controlled conditions since excessively rapid cooling results in fracture of the pellet while excessively slow cooling leads to crystallization of the melt so that the pellet likewise loses its strength.

15 An object of selected embodiments is the provision of a process and an apparatus for producing a pellet preferably intended for subsequent chemical analysis where said process and apparatus are substantially simpler both in terms of process engineering and in terms of apparatus complexity than the prior art processes and apparatuses.

Summary

20 Certain exemplary embodiments provide a process for producing a pellet comprising the steps of: a) melting a material stream to produce a molten material stream; b) shapelessly cooling the molten material stream to obtain a glassy material wherein a grinding step is at least partly effected simultaneously with the cooling thereof, and a ground material stream is produced from the molten material stream; c) pressing at
25 least some of the ground material stream to obtain a pressed pellet.

Other exemplary embodiments provide an apparatus for producing a pellet, said apparatus comprising: a) a melting assembly for melting a material stream to produce

an unshaped molten material stream; b) a cooling assembly for cooling the unshaped molten material stream, wherein the cooling assembly is configured such that a resultant cooled material stream comprises a glassy material, and wherein the cooling assembly comprises rollers or balls and also serves to comminute the unshaped molten material stream; c) a superfine mill for grinding the unshaped molten material stream to produce a ground material stream; and d) a press for producing a pressed pellet from at least some of the ground material stream.

Detailed Description

While in the prior art processes for producing a pellet from a molten material stream the melt is poured into a mold in a defined fashion and cools down therein to afford a pellet, the process disclosed herein comprises shapelessly cooling the molten material stream. Here, the glassy material solidifies to a random shape and is subsequently ground and pressed into a pressed pellet. The rate of this shapeless cooling of the molten material stream is not critical, thus substantially simplifying the entire process.

A glassy material/a glassy structure is to be understood as meaning in particular an amorphous material which after cooling from the melt does not exhibit a crystalline structure. Such a glassy material is obtained, for example, by rapid cooling of the melt to prevent crystallization of the material. The crystallographic properties of the material stream are preferably retained after the cooling of the material stream so that the ground material stream downstream of grinding step c), and the pressed pellet downstream of step d) comprising pressing at least some of the ground material stream into a pressed pellet, have a glassy structure. Neither the grinding of the cooled material stream nor the pressing of the material stream downstream of the grinding alter the crystallographic properties of the material stream and the glassy structure is retained.

The glassy, amorphous structure of the pressed pellet makes subsequent analysis, for example of the composition of the pellet, possible. Moreover, the preceding grinding

and pressing makes such a pellet having a glassy structure significantly more robust than a glassy pellet cooled from the melt.

5 This affords, without shaping the melt but rather from the shapelessly cooled material stream, a pressed pellet which surprisingly yields the same analytical results as are obtained with a conventional fused pellet of the prior art type particularized at the outset. The step of shaping the pellet from the melt, which was hitherto regarded as essential in the art, was found not to be necessary in the experiments underlying selected embodiments. The inventive omission of a complex step of shaping with simultaneous cooling substantially simplifies pellet production both in terms of
10 process engineering and in terms of apparatus.

The process thus combines the techniques of producing a melt, of cooling a melt without complex shaping, and of grinding, with the techniques of pressing a pressed pellet. The novel process allows for substantially simpler handling of the pressed pellet compared to conventional fused pellets in automated environments. A pressed
15 pellet produced in accordance with selected embodiments is moreover markedly more robust than a fused pellet produced by the prior art process. The pressed pellet so produced is highly impervious to handling, in particular by a robot, and does not shatter when correctly prepared.

A further advantage of the process in selected embodiments is the omission of the
20 additional permanent molds (casting molds) made of platinum that are required for the manufacture of the prior art fused pellets.

According to one advantageous embodiment of the process, the shapelessly cooled material stream is ground to a grain size of < 2 mm, preferably < 1 mm.

25 The grinding may also completely or partly be effected simultaneously with the cooling of the molten material stream.

The melt may be homogenized during and/or after the melting operation, preferably by stirring, shaking and/or agitating.

Cooling of the molten material stream may be achieved via a cooling gas and/or a cooling liquid, preferably by means of a metallic surface, for instance a cooled or uncooled metal plate.

5 The grinding advantageously comprises at least a pregrinding and a main grinding, wherein the pregrinding has a duration of more than 30 seconds, preferably more than 150 seconds, in particular more than 280 seconds and wherein the main grinding has a duration of more than 25 seconds and less than 200 seconds. One or more grinding aids may be employed in the pregrinding and/or in the main grinding.

10 The grinding advantageously employs a superfine mill operating at between 650 and 1850 rpm.

According to a further embodiment, the cooling assembly may comprise rollers or balls and may also serve to comminute the unshaped molten material stream.

The cooling assembly may also comprise means that generate a gas and/or liquid curtain through which the unshaped molten material stream passes.

15 The superfine mill may advantageously comprise a vibratory disk mill, a ball mill, a roller mill or a combination of these mill types.

The press may be a ram press.

20 The pressed pellet produced according to the inventive process is generally intended for subsequent chemical analysis. To this end, said pellet is sent to a suitable analytical instrument, preferably an XRF instrument.

The chemical analysis of pressed pellets by x-ray fluorescence analysis (XRF) may in principle be performed on any solid. However, organic substances are rarely considered since they would generally be incinerated at the high temperatures during the melting.

It is generally a mixture of sample material and flux that is melted. The mass ratio between the flux and the sample material is constant for all samples and is preferably 6 :1.

The chemical composition is not changed by the melting operation and cooling.

- 5 The flux renders the melting point independent of the sample material. The melting point of the mixture is generally about 850°C – 1500°C, in particular 1050°C – 1100°C.

Cooling to room temperature generally takes 4-8 minutes.

- 10 To further elucidate selected embodiments, the five steps for producing a pressed pellet via a melt are described hereinbelow.

1. Metering of materials:

- 15 The sample material and a flux (lithium tetraborate, Spectromelt) are metered via a metering apparatus (for example a vibrating chute). The metered amount should be 6-15 g, the ratio between flux and sample material being kept constant for all samples. The mixture is poured into a crucible, preferably made of platinum, graphite, corundum or another suitable ceramics material.

The mixture may be preground prior to melting to achieve improved commixing of sample material and flux.

2. Melting:

- 20 The metered mixture is melted over a gas flame in an oven (for instance an induction oven or muffle oven). To homogenize the melt, a suitable apparatus, in particular a stirring, shaking, or agitating device, may be employed.

3. Cooling:

The melt is rapidly cooled, for instance by one of the following methods:

- pouring the melt onto a cooled metal plate to which an apparatus is connected which detaches the cooled melt from the plate via a mechanical impulse and subsequently cleans said plate. Where necessary, an apparatus for comminuting fused lumps is provided below the metal plate.
- 5
- pouring the melt into a small roller mill ("Mini Polycom"). The rotation of the rollers cools said rollers and the melt.
 - pouring the melt into a cooling liquid.

Where necessary, the relatively coarse particles of the cooled melt are crushed to attain a grain size of < 5 mm.

10 4. Production of a pressed pellet:

The cooled melt is sent to a fine mill. Metered addition is not necessarily required. The grinding may comprise a pregrinding and a main grinding, or a single grinding step. The fine mill operates at between 650 and 1850 rpm.

15 When there is sufficient sample material a presample is defined which is ground in the mill prior to the actual sample and discarded. This avoids any potential contamination by preceding sample material.

Grinding may be performed in the presence of grinding aids.

5. Pressing of the ground material into a pressed pellet.

20 Steps 4 and 5 may be performed using either a combined grinding and pressing device or a fine mill and a pellet press. In the latter case, material is transported from the fine mill to the pellet press via a robot or a conveyor belt for example.

Claims

1. A process for producing a pellet comprising the steps of:
 - a) melting a material stream to produce a molten material stream;
 - b) shapelessly cooling the molten material stream to obtain a glassy material
 5 wherein a grinding step is at least partly effected simultaneously with the cooling thereof, and a ground material stream is produced from the molten material stream;
 - c) pressing at least some of the ground material stream to obtain a pressed pellet.
- 10 2. The process as claimed in claim 1, wherein an unshaped, cooled material stream resulting from the grinding has a grain size of less than 2mm.
3. The process as claimed in claim 1, wherein a step of homogenizing is carried out during, or after the melting operation, or both.
- 15 4. The process as claimed in claim 1, wherein the cooling of the molten material stream is achieved via a cooling gas, a cooling liquid, a cooled or uncooled metallic surface, or a combination thereof.
5. The process as claimed in claim 1, wherein the grinding step comprises at least a pregrinding and a main grinding, wherein the pregrinding has a duration of more than 30 seconds.
- 20 6. The process as claimed in claim 1, wherein the grinding step employs a superfine mill operating at between 650 and 1850 rpm.
7. The process as claimed in claim 1, wherein the ground material stream is fed to a pressing device.

8. The process as claimed in claim 5, wherein a grinding aid is employed in the pregrinding, the main grinding, or both.
9. An apparatus for producing a pellet, said apparatus comprising:
- 5 a) a melting assembly for melting a material stream to produce an unshaped molten material stream;
- 10 b) a cooling assembly for cooling the unshaped molten material stream, wherein the cooling assembly is configured such that a resultant cooled material stream comprises a glassy material, and wherein the cooling assembly comprises rollers or balls and also serves to comminute the unshaped molten material stream;
- c) a superfine mill for grinding the unshaped molten material stream to produce a ground material stream; and
- d) a press for producing a pressed pellet from at least some of the ground material stream.
- 15 10. The apparatus as claimed in claim 9, wherein the cooling assembly comprises a cooled or uncooled metal plate.
11. The apparatus as claimed in claim 9, wherein the cooling assembly comprises means that generate a gas curtain, a liquid curtain, or a combination thereof.
- 20 12. The apparatus as claimed in claim 9, wherein the superfine mill comprises a vibratory disk mill, a ball mill, a roller mill or a combination of these mill types.
13. The apparatus as claimed in claim 9, wherein the press is a ram press.
14. The process as claimed in claim 1, wherein the pellet is intended for subsequent analysis to identify substances.
- 25 15. The process as claimed in claim 2, wherein the material stream is ground to a grain size of less than 1mm.

16. The process as claimed in claim 3, wherein the homogenizing is carried out by stirring, shaking or a combination thereof.
17. The process as claimed in claim 5, wherein the pregrinding has a duration of more than 150 seconds.
- 5 18. The process as claimed in claim 5, wherein the pregrinding has a duration of more than 280 seconds.
19. The process as claimed in claim 5, wherein the main grinding has a duration of more than 25 seconds and less than 200 seconds.
- 10 20. The apparatus as claimed in claim 9, wherein the pellet is intended for subsequent analysis to identify substances.