



METHOD FOR DIRECTLY COOLING FINE-PARTICLE SOLID SUBSTANCES

- 5 The present invention relates to a method and a device for direct cooling of fine-particle, pulverulent solid substances prior to cooling in bulk material packings in accordance with the preamble of Claims 1 and 23.

- Countless, pulverulent solid substances are filled in bulk material packings for the purpose of sales, transport, and protection from environmental influences or for the purpose of storage. The temperature of fine-particle, pulverulent solid substances is frequently too high for trouble-free filling into desired bulk material packings on account of the production or processing procedures. Without adequate cooling this can accordingly lead to innumerable problems or drawbacks, such as e.g.
- 15 - pronounced wear by temperature-sensitive plant components such as e.g. rubber seals or compression valves
 - damage to bulk material packings, such as e.g. paper bags or plastic bags, from excessively high temperature of solid substances
 - low bulk weight of solid substances at time of filling
 - 20 - poor ventilation during filling of bulk material packings and silo vehicles due to comparatively low viscosity of the hot gas phase
 - low fill quantity of bulk material packings and silo vehicles
 - more time-efficient filling of bulk material packings and silo vehicles
 - damage to bulk material packings (e.g. paper bags) from excessively high filling
 - 25 volume
 - strong tendency to dust during filling of solid substances in bulk material packings
 - optically less responsive appearance of bags and pallets
 - condensation effects inside pallets with shrink cap
 - impairment of work safety with further handling (e.g. unloading or transportation)
 - 30 through hot surface of bulk material packings.

- The abovementioned problems or disadvantages during filling of fine-particle, pulverulent solid substances in bulk material packings can occur both individually and in combination. Some of the abovementioned problems or disadvantages are anticipated when hot solid substances have to be handled and filled in bulk material packings, while other of the abovementioned problems or disadvantages
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(e.g. low bulk weight, poor ventilation) however are specific for the handling and filling of very fine-particle, hot, pulverulent solid substances in bulk material packings. In this sense such pulverulent solid substances are designated in the present document as fine-particle, and have an average particle size of less than
5 50 μm , preferably less than 20 μm , but in particular such solid substances, which have an average particle size of less than 5 μm , preferably less than 1 μm .

Bulk material packings in the present document are understood to mean those packings, which are used for the purpose of sale, of transport, protection from
10 environmental influences or for the purpose of long-term storage, for example paper bags or plastic bags, barrels, big bags, sacks, containers made of paper, pulp, plastic or other materials.

Basically such problems can arise during filling in bulk material packings with all
15 fine-particle, pulverulent solid substances, which have a higher temperature resulting from the type of manufacture or processing, e.g. following calcining, calcining with subsequent milling, drying, other high-temperature processing or in a manufacturing procedure at high temperature.

20 By way of example this applies to cement, soot or to pigments pre-treated thermally, e.g. by calcining, which are to be filled in bulk material packings. But in particular this applies to steam jet-milled pigments.

For milling of very fine-particle solid substances with particle sizes in a range of 1
25 μm or under the technique of jet milling is frequently used. Jet milling is impact milling, wherein the milling goods are accelerated in a rapid gas flow and comminuted by impacting one another or against an impact wall. The milling effect here depends decisively on impact velocity or impact energy.

30 With milling of solid substances, for the comminution or disagglomeration of which a particularly high power supply or a particularly high degree of separation is required, such as e.g. for different inorganic pigments, usually the method of steam jet milling is used with superheated steam as milling medium. A method for manufacturing titanium dioxide pigments, in which the titanium dioxide is finally
35 subjected to steam jet milling, is described in DE 195 36 657 A1.

In the case of steam jet milling the temperature of the pigment-gas mixture after it leaves the mill is mostly in the range of ca. 200 to 300°C. After the pigment is separated from the water vapour by means of cyclone or dust filter the temperature of the bulk pigment containing water vapour is typically still in the range of ca. 200
5 to 250°C. Due to interim storage in silos or during mechanical or pneumatic conveying a certain cooling still sets in, but at the time of filling bags or other packings the pigments frequently still display temperatures of around 100°C or above. This can lead to countless problems or disadvantages, as already described hereinabove.

10 The cooling effect of fine-particle, pulverulent solid substances through addition of air of ambient temperature, e.g. during pneumatic conveying of the fine-particle, pulverulent solid substances, is confined to the air on account of the minimal temperature difference and the minimal heat capacity. The result of this is that
15 even when large quantities of air are used the drop in temperature is comparatively slight and accordingly the abovementioned disadvantages are defrayed only mildly.

A very large quantity of air can indeed cause a significant drop in temperature, yet
20 this method has considerable disadvantages with respect to separation of the gas phase from the fine-particle, pulverulent solid substances as well as with respect to filling properties and operating costs. In addition, the danger of contamination of the fine-particle, pulverulent solid substances through humidity, dust, carbon dioxide, sulphur oxide or nitric oxide, oil traces or other contaminants from the air.

25 Convection cooling over a longer period is likewise disadvantageous because of the poor thermal conductivity of most of the fine-particle, pulverulent solid substances and accordingly disproportionately long blocking of bulk goods containers. This can lead to a significant reduction in production capacity.

30 DE 3 414 035 A1 describes the indirect cooling of a gas containing dust, in which the gas is conveyed via regenerators filled with a heat storage medium.

35 These methods or comparable methods for indirect cooling via cooling faces or heat exchangers are little effective, as considerable heat transfer surfaces would have to be provided for this purpose. With this method described in DE 3 414 035 A1 the dust loading of the gases with ca. 20 mg/m³ is very slight and not

comparable with the solid-gas mixture ratios usual e.g. in pigment technology with intentional filling in bulk material packings (frequently several kg/m³). From experience, in the case of higher solid contents in the gas phase the danger of sticking on the cooling contact surfaces increases.

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The use of cooling screws for indirect cooling of fine-particle, pulverulent solid substances is applied in various ways, yet here too the disadvantages with respect to sticking on, operating safety and investment costs are considerable.

10 DE 3713571 AI describes a device for filling plastic bags with pulverulent or granulated materials, whereby the filled bags are welded close and in brought to a cooling zone to ensure stability of the welded seam.

15 Since the cooling described here is carried out only after filling in bags, it is not suitable for avoiding the abovementioned disadvantages.

US 3 664 385 describes mechanical compacting of powders for filling of packings, whereby a pulse of cooled air can also be used for dust removal.

20 The cooling effect of such a pulse of cooled air however is not sufficient to achieve adequate compacting of fine-particle, pulverulent solid substances, which is why the powders still have to be compacted mechanically. Besides that, this method for fine-particle, pulverulent solid substances with special requirements with respect to dispersibility and fine particle capacity, such as e.g. pigments, is less suited,
25 because there is the danger of agglomeration as a result of mechanical compacting and with that the purpose intended by milling can be partly eliminated.

US 4619113 describes direct cooling of washing powder in a silo with the aid of volatile nitrogen, in order to then enable the addition of temperature-sensitive
30 washing powder additives.

Since the average particle size of the washing powder to be cooled is 500 µm (column 1, lines 24-25), this method described in US 4619113 offers neither a reference to the special problems with filling of very fine-particle, pulverulent solid
35 substances in bulk material packings, nor their solution.

DE 3941262 describes direct cooling of a pulverulent substance by means of volatile nitrogen, whereby the stream of powder is scattered by mechanical baffles before it makes contact with the volatile nitrogen.

- 5 DE 3623724 A1 describes direct cooling of cement by means of volatile nitrogen, whereby the cement is blasted with the volatile nitrogen into a cement silo at the same time.

10 These methods described in DE 3941262 and DE 3623724 A1 cause exclusively simple cooling, but not an improvement in the filling properties for packaging in bulk material packings. These documents neither make reference to the specific requirements of very fine-particle pulverulent solid substances, nor make reference to the abovementioned specific problems and their solution in handling and filling of fine-particle, pulverulent solid substances in bulk material packings.

15 US 3330046 describes the heat exchange between gases and fine-particle solid substances with a particle size of less than 50 μm , whereby a device comprising several interconnected chambers is used, through which the gas and solids flow occurs in the opposite direction.

20 The plant used for this is expensive and a large quantity of cooling gas is required. The method accordingly has similar disadvantages to the abovedescribed cooling with large quantities of air. Therefore there are no references made to the abovementioned specific problems and their solution in handling and filling of fine-
25 particle, pulverulent solid substances in bulk material packings.

EP611 928 A1, EP501 495 A1 and DE 38 33 830 A1 describe direct cooling of milled goods with low-boiling, condensed gases. But this cooling takes place before the milling to optimise the actual milling process by increasing the friability
30 of the milled goods and does not consequently lead to a solution of the abovedescribed problems.

The drop in temperature caused by such cooling prior to milling is generally lost again through the development of heat associated with the milling, so that there
35 are no more advantages during filling.

There are also no references to the abovementioned specific problems and their solution in handling and filling of fine-particle, pulverulent solid substances in bulk material packings.

- 5 The aim of most of the abovementioned methods is to reduce obvious thermal consequences by different variants of cooling. Explicit references to the specific requisite of fine-particle, pulverulent solid substances, e.g. the very high requirements with respect to fine particle capacity and dispersibility of pigments, and the problems associated with filling in bulk material packings are not to be
10 found here.

The drawback to the abovementioned methods is that they do not eliminate or only
yo so to a minor extent the abovedescribed problems with handling and filling of
fine-particle, pulverulent solid substances in bulk material packings. This applies in
15 particular to the foregoing pigment-specific problems.

The object was to provide a method, which enables a hot, fine-particle, pulverulent
solid to be processed such that the abovedescribed disadvantages with the
handling of fine-particle, pulverulent solid substances and with subsequent filling in
20 bulk material packings are avoided either fully or at least extensively.

Another object was to use the least possible gas volume during handling of fine-
particle, pulverulent solid substances and during filling in bulk material packings in
order to keep expenditure during separation of the gas and dust extraction as low
25 as possible.

In addition to that, it was an object to provide a device, with which a mixture
comprising a fine-particle, pulverulent solid and a gas can be cooled in a simple
and efficient manner.
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And it was also an object to produce fine-particle, pulverulent solid substances
present in bulk material packings with a smaller proportion of oxygen as compared
to air in the gas phase between the solid particles.

35 A method and a device in accordance with Claims 1 and 23 solve this task.

The inventive method comprises direct cooling of fine-particle, pulverulent solid substances by addition of a cooling agent and following this filling the milled goods in bulk material packings, whereby the cooling agent comprises either one or more different low-boiling, condensed gases, or consists of a cold gas or gas mixture, which was generated by means of one or more different low-boiling, condensed gases, or whereby the cooling agent comprises a cold gas or gas mixture, which was pre-cooled by means of one or more different low-boiling, condensed gases. This method is of particular significance for cooling and ensuing filling of fine-particle, pulverulent solid substances in bulk material packings, such as e.g. commercially available paper bags, plastic bags, sacks, barrels or other small containers made of a variety of materials. The method can also be beneficial with filling silo vehicles.

All those compounds, which have inert behaviour with respect to the fine-particle, pulverulent solid substances in question, are suitable as cooling agent. These can be for example inert gases, carbon dioxide, nitrogen, oxygen or mixtures of the abovementioned substances (e.g. air).

The cooling agent used for direct cooling is preferably obtained from vaporising low-boiling, condensed gases. Volatile nitrogen, volatile air or volatile carbon dioxide are particularly suitable. Solid carbon dioxide is also suitable, particularly when it is in finely distributed form.

The cooling agent used for the direct cooling preferably has a temperature of less than 0°C, preferably less than -20°C, particularly preferably less than -40°C, to cause a significant cooling effect.

The desired temperature for the fine-particle, pulverulent solid substances to be cooled or the mixture containing solids to be cooled naturally depends on the type of solid substances and the quality requirements of these. The type and quantity of the cooling agent is preferably selected such that the fine-particle, pulverulent solid substances to be cooled by at least 20°C, are preferably cooled by at least 50°C. Cooling via direct cooling is preferred at a maximum 100°C, particularly preferably at a maximum 70°C.

The cooling agent used for direct cooling contains preferably less than 0.0001 mass fraction of water. In this way, the dew point of the solid-gas mixture can be

lowered, which precipitates to form agglomerates with diminished tendency to solid substances. By way of example the dew point can be lowered advantageously in the bulk solid by using volatile nitrogen with its extremely minimal water content.

- 5 Cooling of the fine-particle, pulverulent solid substances can in principle take place at different points of the manufacturing or processing procedure, for example directly following thermal treatment of the pulverulent solid substances, in feed pipes or directly prior to filling in bulk material packings.
- 10 The inventive method is particularly effective if the fine-particle, pulverulent solid substances are created or processed at high temperatures on account of process-technological or logistical conditions and the temperature can be lowered only inadequately by convection cooling or cooling with air, so that conventional filling in bulk material packings attracts considerable problems.
- 15 Examples for processes, in which the inventive cooling can have an advantageous effect:
- direct cooling and filling of fine-particle, pulverulent solid substances following thermal drying and possibly milling
 - 20 - direct cooling and filling of fine-particle, pulverulent solid substances following calcining and optionally milling
 - direct cooling and filling of fine-particle, pulverulent solid substances following a pyrolytic manufacturing process or a combustion process
 - direct cooling and filling of fine-particle, pulverulent solid substances following
 - 25 steam jet milling.

It has been shown that with fine-particle pulverulent solid substances having an average particle size of $< 50 \mu\text{m}$, preferably $< 20 \mu\text{m}$, but in particular with very fine-particle $< \text{RTI}$ pulverulent solid substances having an average particle size of

30 $< 5 \mu\text{m}$, particularly preferably $< 1 \mu\text{m}$, the rheologic properties or the handling properties depend clearly on their temperature. So for example the bulk weight of fine-particle, pulverulent solid substances, which are filled at low temperature in the customarily used packings such as bags, barrels, big bags or silos, is significantly higher compared to the same product, which are filled at higher temperature in

35 these packings. These fine-particle, pulverulent solid substances to be cooled and filled can for example be titanium dioxide, iron oxide, chromium oxide, fade-proof

pigments, coloured pigments, metallic pigments, magnetic pigments, soot or cement.

5 The fine-particle, pulverulent solid substances to be filled can also be temperature-sensitive compounds or solid substances coated with temperature-sensitive compounds.

10 The inventive direct cooling by a cooling agent can for example be carried out by the cooling agent being metered into a feed pipe for pneumatic feeding of the fine-particle, pulverulent solid substances. It can also be advantageous to add the cooling agent in at several different points, particularly to achieve a sharp drop in temperature. In this way the cooling agent can optionally also serve as a substantial contribution to pneumatic conveying.

15 The cooling agent can be injected into a supply pipe both in the direction of flow and also against the direction of flow, depending on whether strong or slight turbulence of the cooling agent is aimed for.

20 The device for inventive direct cooling comprises a reservoir for low-boiling, condensed gases, an insulated compound pipe between reservoir and supply pipe for pneumatic conveying of the fine-particle, pulverulent solid substances, a nozzle for injecting the low-boiling, condensed gases into the supply pipe and a control and regulating mechanism.

25 The advantage of this method of inventive direct cooling over the method of indirect cooling via contact surfaces is that no large contact surfaces are required for heat transfer. In addition, the cooling takes place substantially faster than with indirect cooling, which has particularly advantageous effect, if there are temperature-sensitive milled goods involved or when large drops in temperature
30 are targeted in a short period.

By way of comparison, with indirect cooling there is the risk of localised condensation, in particular in the region of the cooling surfaces on account of comparatively high temperature gradients in the material to be cooled.

35 The advantage of the inventive method as compared to cooling with large quantities of air from the ambient temperature on the one hand is that a

substantially faster and stronger cooling effect can be achieved from direct cooling using low-boiling, condensed gases on account of their vaporising enthalpy.

5 On the other hand with the inventive method a smaller volume of gas phase is required, which substantially facilitates separation of the gas phase from the fine-particle, pulverulent solid substances prior to or during filling of bulk material packings. Furthermore, the cost of dust removal from the gas is considerably diminished.

10 Apart from that contaminating of the fine-particle, pulverulent solid substances by humidity, dust, carbon dioxide, sulphur oxide or nitric oxide, oil traces or other contaminants from the air is reduced or avoided. In particular, in the case of a basic surface of fine-particle, pulverulent solid substances (e.g. after chemical treatment with corresponding compounds) undesired neutralising of the surface by
15 the acidic constituents in air could occur when large quantities of air are used.

Finally, the residual moistness of the fine-particle, pulverulent solid substances is reduced by the absorbability of humidity of the generally very dry cooling agent.

20 Although cryogenic cooling of fine-particle, pulverulent solid substances by means of low-boiling, condensed gases is distinguished by relatively high costs for the cooling agent, on closer observation this effect is surprisingly overcompensated by a whole range of advantages of this method of operation. By way of example the following advantages can result from the lower temperature of the solid-gas
25 mixture and from the lesser specific proportion of gas (relative to solids):

- less wear by temperature-sensitive plant parts such as e.g. rubber seals
- less damage to temperature-sensitive bulk material packings such as e.g. paper bags, plastic bags because of lower temperature of the fine-particle, pulverulent solid substances to be filled
- 30 - the option of using more cost-efficient packings because of less thermal load
- higher bulk weight of fine-particle, pulverulent solid substances at the time of filling
- better ventilation during filling of bulk material packings and silo vehicles on account of the lower viscosity of the gas phase
- 35 - higher fill quantity of bulk material packings and silo vehicles
- faster filling of bulk material packings and silo vehicles

- less damage to bulk material packings (e.g. paper bags) from excessively high filling volume
- less tendency to dust during filling of fine-particle, pulverulent solid substances in bulk material packings
- 5 - less gas volume in pneumatic conveying or cooling; therefore smaller filter surface for dust removal from the gas or higher filling capacity with constant filter surface
- optically more responsive appearance, for example of bags and pallets
- less humidity in the product through absorbability of the cooling agent
- 10 - less condensation inside pallets with shrink cap
- improvement in work safety through lower temperature of the packaged fine-particle, pulverulent solid substances during subsequent handling (e.g. loading or transportation)

15 The consequence of the inventive method for cooling fine-particle, pulverulent solid substances is not only a lower proportion of faulty charges, such as e.g. burst bags, but also has the added advantages of more reliable operation, increased capacity of filling mechanisms as well as a lesser tendency to dust, and results in a more responsive optical appearance of individual bags or pallets.

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The inventive method is particularly suited to direct cooling of inorganic or organic pigments and their subsequent filling in bulk material packings, because particularly high demands are often placed on these pigments with respect to dispersing or fineness, and at the same time the rheologic properties or the

25 handling of the ground pigments are of particular significance.

The specific advantages of the inventive method when applied to pigments are that substantial demands on the pigments, such as good dispersibility, good optical properties and good handling of ground pigments during filling in bulk material

30 packings, can be fulfilled at the same time. The rheologic properties or the handling properties of pigments depend considerably on their temperature. So, for example, the bulk weight of pigments, which are filled at lower temperature in the normally used packings such as bags, barrels, big bags or silos, is significantly higher compared to the same product, which are filled at higher temperature in

35 these packings.

In steam jet milling of pigments the cooling of the milled goods can take place, for example, directly behind the steam jet mill, after a separation device such as e.g. a dust filter or a cyclone in supply pipes or immediately prior to filling in bulk material packings. It can be advantageous if the cooling agent is added only when the partial water vapour pressure of the gas phase of the milled goods for example already has adequately low values due to partial exchange or dilution of the water vapour by air, in order to avoid the dew point falling below as a result of direct cooling.

Preferably after the steam jet milling a portion of the gas phase containing water vapour is first separated off from the ground pigments and then the ground pigments are cooled via direct cooling by means of a cooling agent. This separating of a portion of the gas phase containing water vapour is carried out for example by means of a cyclone and/or a dust filter.

The inventive method is suited in particular to titanium dioxide pigments due to the strong influence of temperature on parameters such as the bulk weight during filling. In particular, the inventive method proves to be particularly advantageous for such titanium dioxide qualities, which are used for dyeing plastic materials or for dispersion dyes and on account of their specific composition have a particularly low bulk weight. In this way alternative methods such as e.g. granulation or pelleting can be dispensed with.

Also, a disadvantageous influence on the tendency to disintegration of organic additives on the pigment surface (milling agents) can easily arise with organically coated pigments from high temperatures, which can in particular have a negative effect on the shade of colour.

This is likewise avoided by the inventive method.

The inventive method is particularly beneficial if several steam jet mills are operated in parallel and the high pigment throughput achieved in this way can be lowered in temperature only inadequately through convection cooling or other conventional methods.

The inventive method is also well suited to iron oxide pigments. The advantages here are similar as for titanium dioxide pigments. With oxidisable iron oxide

pigments, e.g. iron oxide pigments in magnetite modification, there is also the fact that through using non-oxidising gases such as e.g. CO₂ or N₂ as cooling agent the tendency to oxidation during the course of processing and storing can be prevented by the partial or extensive displacement of atmospheric oxygen by the cooling agent. In addition, the reactivity of these pigments as compared to residual portions of atmospheric oxygen is clearly decreased through low temperatures, as achieved by the inventive method during filling in bags.

By means of the inventive method it is thus possible in many cases to also comminute such pigments, which usually cannot be ground by means of steam jet mills, with this extremely efficacious milling technique.

The materials placed in bulk material packings according to the present invention have, in the gas phase between the solid particles, preferably less than 20% by weight oxygen, particularly preferably less than 15% by weight oxygen or a mass ratio of nitrogen to oxygen of more than 4.7, preferably more than 5.7.

Because fine-particle, pulverulent solid substances can show clearly differing rheologic behaviour depending on specific composition, focussed influencing of the conveying properties can be undertaken through appropriate selection of the addition of the cooling agent and thus the temperature. And depending on the type of solid cooling prior to pneumatic or mechanical conveying or only after conveying can be more beneficial. Also, cooling immediately prior to filling in bulk material packings can be an advantage.

A preferred embodiment of the invention is to cool the conveying air used for pneumatic conveying and to carry out direct cooling of the fine-particle, pulverulent solid substances using this cold conveying air as cooling agent. The temperature of the conveying air can be selected such that after being mixed with the fine-particle, pulverulent solid substances to be cooled the desired final temperature is reached. Cooling of the conveying air can be done for example by means of a heat exchanger or via direct supply of low-boiling, condensed gases or solid carbon dioxide in the conveying air.

Cooling of the conveying air by means of heat exchanger can be carried out in accordance with any method familiar to the expert. It is particularly advantageous

with this method of operation that indirect cooling takes place here in a gas free of solids and that the solid-gas mixture is cooled directly.

5 Cooling of the conveying air used for pneumatic conveying prior to contact with the fine-particle, pulverulent solid substances to be cooled can be more favourable in process technology terms than cooling of a gas containing solids, such as occurs after mixing the conveying air with the fine-particle, pulverulent solid substances to be conveyed.

10 It is also possible to combine these variants together. By way of example, both cooling of the conveying air (directly or indirectly) and direct cooling of the solid-gas mixture with the latter can take place, and additionally direct cooling of the solid-gas mixture (e.g. with low-boiling, condensed gases) can be undertaken.

15 Independently of the type of cooling agent in the case of the inventive method of direct cooling of fine-particle, pulverulent solid substances the option of focussed control and regulation of the overall process with respect to temperature-dependent parameters is particularly advantageous. By way of example, an optimal and constant temperature of the fine-particle, pulverulent solid substances
20 can be adjusted by regulating the additional quantity or temperature of the cooling agent, depending on the product, and thus e.g. the conveying properties or the properties for filling bulk material packings can be precisely controlled, optimised and kept constant.

25 The fine-particle, pulverulent solid substances are usually filled in paper bags or plastic bags via a filling silo by means of solids feed systems. At temperatures of 60°C and over the use of cost-efficient plastic bags (e.g. those made out of polyethylene or polypropylene) is generally ruled out. Only special and expensive plastic bags with high temperature stability can be used for this purpose.

30 By means of the inventive method it is possible, however, to make use of cost-effective polyethylene or polypropylene bags, instead of these costly plastic bags. For this, cooling at temperatures in the region of 60°C or less is generally required.

35 The use of more cost-effective paper bags with a lower stability, or paper bags with a synthetic lining or synthetic components in the region of wear is feasible by means of the inventive method.

The inventive method also enables the use of other packing means, which are not durable compared to the temperatures occurring according to the prior art, e.g. big bags based on plastics with minimal thermal load capacity or other temperature-sensitive plastic packaging.

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Example 1: A post-treated and dried titanium dioxide pigment manufactured according to the prior art is micronised in a steam jet mill.

10 The product-steam ratio is 1 : 2.2 parts by weight. The temperature of the steam is 260°C. Ca. 100 m³ per tonne TiO₂ conveying air together with the pigment to be ground are introduced into the steam jet mill. The temperature of the pigment-gas mixture after the steam jet milling is ca. 230°C; after the pigment is separated off from the gas phase by means of a cyclone the temperature of the bulk pigment containing water vapour is ca. 180°C. The water content of the gas phase is ca.
15 95% by weight, the water content of the gas phase relative to TiO₂ is ca. 0.2% by weight with a bulk density of TiO₂ of 0.5g/cm³.

Following interim storage in a silo the bulk pigment containing water vapour is conveyed pneumatically to the bagging machine. The quantity of dried conveying
20 air is 75 m³ per tonne TiO₂. Ninety-seven l liquid nitrogen per tonne TiO₂ is introduced to the supply pipe via a nozzle. The temperature of the titanium dioxide-gas mixture is hereby lowered from 110°C to 60°C. The water content of the gas phase is accordingly ca. 1% by weight, while the water content of the gas phase relative to TiO₂ is ca. 0.2%.

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The gas phase is separated from the TiO₂ in the filling silo, from which the bags are filled. The temperature of the gas-containing bulk pigment at the time of filling in paper bags is 60°C. The composition of the gas phase between the TiO₂ particles in the paper bags is: 87% by weight N₂, and 2% by weight O₂. The water
30 content of the gas phase relative to TiO₂ is calculated at less than 0.01% by weight. Independently of this the TiO₂ has approximately 0.3% by weight of adsorbed water.

35 The filling and ventilation behaviour during filling of the bags is good due to the high bulk weight of the gaseous bulk pigment. As a consequence the pallets have an optically responsive appearance.

Example 2: A post-treated and dried titanium dioxide pigment manufactured according to the prior art for dyeing plastics is micronised in a steam jet mill with the addition of 1% by weight of a silicon oil. The further sequence of the method up to filling in bags follows that in Example 1.

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In contrast to Example 1 the filling is done into commercially available polypropylene bags.

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On account of the low temperature of the gaseous bulk pigment (60°C) at the time of filling in the polypropylene bags there is no damage at all to the bags.

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The filling and ventilation behaviour during filling of the bags is good due to the comparatively high bulk weight of the gaseous bulk pigment. As a consequence the pallets have an optically responsive appearance.

The pigment is particularly suited to dyeing plastics. It is possible to utilise the pigment together with the packaging directly in the treatment process.

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Example 3: A titanium dioxide pigment produced according to the prior art and post-treated and dried with overall 15% by weight of SiO_2 and Al_2O_3 (relative to TiO_2) is micronised in a steam jet mill. The further sequence of the method follows that in Example 1.

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The filling and ventilation behaviour during filling the bags is good due to the comparatively high bulk weight of the gaseous bulk pigment. As a consequence the pallets have an optically responsive appearance. The pigment is particularly suited to the production of dispersion dyes.

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Example 4: An iron oxide red pigment (Fe_2O_3) produced according to the prior art by calcining from magnetite is micronised in a steam jet mill. The product-steam ratio is 1 : 2 parts by weight. The temperature of the steam is 260°C. The further sequence of the method follows that in Example 1.

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The filling and ventilation behaviour during filling of the bags is good on account of the high bulk weight of the gaseous bulk pigment. Also, it is possible to fill the product in conventional polyethylene or polypropylene bags due to the low temperature (60°C) of the gaseous bulk pigment.

Example 5: A nickel rutile yellow pigment produced according to the prior art by calcining is ground in a Raymond mill. Following interim storage in a silo the bulk pigment is conveyed pneumatically to the bagging machine. Sufficient liquid nitrogen is introduced into the supply pipe via a nozzle for the temperature of the gaseous bulk pigment at the time of filling in bags to be 60°C.

The filling and ventilation behaviour during filling of the bags is good on account of the high bulk weight of the gaseous bulk pigment. Also, it is possible to fill the product in conventional polyethylene or polypropylene bags due to the low temperature (60°C) of the gaseous bulk pigment.

Example 6: Furnace soot produced according to the prior art is separated using a dust filter from the gas phase and is interim-stored in a silo. From there the soot is fed pneumatically to the bagging machine. Sufficient liquid nitrogen is introduced into the supply pipe via a nozzle for the temperature of the gaseous bulk soot at the time of filling in bags to be 60°C.

The filling and ventilation behaviour during filling of the bags is good on account of the high bulk weight of the gaseous bulk soot. Also, it is possible to fill the product in conventional polyethylene or polypropylene bags due to the low temperature (60°C) of the gaseous bulk soot.

Example 7: A magnetite pigment (Fe_3O_4) produced according to the prior art is ground in a steam jet mill. After extensive separation of the gas phase by means of a cyclone or dust filter and interim storage in a silo the bulk pigment is pneumatically conveyed with nitrogen as conveying gas to the bagging machine. Sufficient liquid nitrogen is introduced into the supply pipe via a nozzle for the temperature of the gaseous bulk pigment at the time of filling in bags to be a maximum of 30°C.

The filling and ventilation behaviour during filling of the bags is good on account of the high bulk weight of the gaseous bulk pigment. Reactivity relative to residual parts of oxygen is prevented on account of the low filling temperature.

The jet stream-milled magnetite exhibits excellent fine particle capacity and dispersibility.

Example 8 (comparative example): A post-treated and dried titanium dioxide pigment produced according to the prior art is micronised in a steam jet mill.

5 The product-steam ratio is 1 : 2.2 parts by weight. The temperature of the steam is 260 C. Ca. 100 m³ per tonne TiO₂ conveying air together with the pigment to be milled is introduced into the steam jet mill. The temperature of the pigment-gas mixture after steam jet milling is ca.230°C; after separation of the pigment from the gas phase by means of a cyclone the temperature of the bulk pigment containing water vapour is ca. 180°C. The water content of the gas phase is ca. 95% by weight, while the water content of the gas phase relative to TiO₂ is ca. 0.2% by weight at a bulk density of TiO₂ of 0.5g/cm³.

Following interim storage in a silo the bulk pigment containing water vapour is conveyed pneumatically to the bagging machine. The quantity of conveying air is 15 150 m³ per tonne TiO₂.

The water content of the gas phase is ca. 1% by weight, while the water content of the gas phase relative to <RTI TiO₂ is ca. 0.2%.

20 The gas phase is separated from the TiO₂ in the filling silo, from which the bags are filled. The temperature of the gaseous bulk pigment at the time of filling in paper bags is 110°C. The composition of the gas phase between the TiO₂ particles in the paper bags is: 76% by weight N₂, 23% by weight O₂.

25 The filling and ventilation behaviour during filling of the bags is poor due to the low bulk weight of the gaseous bulk pigment. As a consequence the pallets have an optically less responsive appearance.

Filling in commercially available polyethylene or polypropylene bags is not possible on account of the high temperature. 30

Example 9 (comparative example): A post-treated and dried titanium dioxide pigment produced according to the prior art is micronised in a steam jet mill.

35 The product-steam ratio is 1 : 2.2 parts by weight. The temperature of the steam is 260°C. Ca. 100 m³ per tonne TiO₂ conveying air are introduced together with the pigment to be ground to the steam jet mill. The temperature of the pigment-gas

- mixture after the steam jet milling is ca. 230°C; after the pigment is separated from the gas phase by means of a cyclone the temperature of the bulk pigment containing water vapour is ca.180°C. Following brief interim storage in a silo the bulk pigment containing water vapour is conveyed pneumatically to the bagging
- 5 machine. To achieve sufficient cooling of the titanium dioxide, 730 m³ of conveying air per tonne TiO₂ are used. The gas phase is separated from the TiO₂ in the filling silo, from which the bags are filled. The temperature of the gaseous bulk pigment at the time of filling in paper bags is 60°C.
- 10 Separation of the gas phase is complicated on account of the large quantity of gas. Only considerably less titanium dioxide per time unit can be delivered and filled, if no correspondingly enlarged filter surfaces are available for removing dust from the conveyor gas.

Claims:

1. A method for direct cooling of fine-particle, pulverulent solid substances by
5 addition of a cooling agent and subsequent filling in bulk material packings,
whereby the fine-particle solid substances have an average particle size of less
than 50 μm , preferably less than 20 μm , and whereby the cooling agent comprises
a) either one or more different low-boiling, condensed gases
b) or a cold gas or gas mixture, which is created by means of one or more different
10 low-boiling, condensed gases
c) or a cold gas or gas mixture, which is pre-cooled by means of one or more
different low-boiling, condensed gases.
2. The method as claimed in Claim 1, characterised in that the fine-particle,
15 pulverulent solid substances have an average particle size of less than 5 μm ,
preferably less than 1 μm .
3. The method as claimed in Claim 1, characterised in that the fine-particle,
pulverulent solid substances to be cooled and filled are inorganic or organic
20 pigments.
4. The method as claimed in Claim 1, characterised in that the fine-particle,
pulverulent solid substances to be cooled and filled can be titanium dioxide, iron
oxide, chromium oxide, fade-proof pigments, coloured pigments, metallic pigments,
25 magnetic pigments or soot.
5. The method as claimed in Claim 1, characterised in that the fine-particle,
pulverulent solid to be cooled and filled is cement.
- 30 6. The method as claimed in Claim 1, characterised in that the fine-particle,
pulverulent solid substances are temperature-sensitive compounds or solid
substances coated with temperature-sensitive compounds.
7. The method as claimed in Claim 1, characterised in that the fine-particle,
35 pulverulent solid substances are milled prior to the procedural step of direct
cooling.

8. The method as claimed in Claim 1, characterised in that the fine-particle, pulverulent solid substances are pigments, which are cooled directly by addition of a cooling agent between the procedural step of steam jet milling and the procedural step of filling in bulk material packings or mobile bulk goods containers.

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9. The method as claimed in Claim 8, characterised in that of the mixture of ground pigment and gas phase containing water vapour present immediately after steam jet milling a part of the gas phase containing water vapour is first separated from the ground pigments by means of a cyclone and/or a dust filter and then the ground pigments are cooled by direct cooling by means of a cooling agent.

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10. The method as claimed in Claim 1, characterised in that the cooling agent used for direct cooling has a temperature of less than 0°C, preferably less than -20°C, particularly preferably less than -40°C.

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11. The method as claimed in Claim 1, characterised in that the fine-particle, pulverulent solid substances for filling in bulk material packings or mobile bulk goods containers are conveyed pneumatically and the gas used for the pneumatic conveying is used as cooling agent for direct cooling, whereby this gas is cooled prior to contact with the fine-particle, pulverulent solid substances either indirectly in a heat exchanger by means of low-boiling, condensed gases or directly by injection of one or more different low-boiling, condensed gases.

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12. The method as claimed in Claim 1, characterised in that the cooling agent is added in to the supply pipe during pneumatic conveying of the fine-particle, pulverulent solid substances.

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13. The method as claimed in Claim 1, characterised in that gaseous nitrogen or gaseous carbon dioxide is used as cooling agent.

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14. The method as claimed in Claim 1, characterised in that liquid nitrogen or liquid or solid carbon dioxide is used as cooling agent.

15. The method as claimed in Claim 1, characterised in that the fine-particle, pulverulent solid substances are cooled within the brief period, required for mixing with the cooling agent, by at least 20°C, preferably by at least 50°C.

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16. The method as claimed in Claim 1, characterised in that the fine-particle, pulverulent solid substances are cooled to maximum 100°C, preferably to maximum 70°C.
- 5 17. The method as claimed in Claim 1, characterised in that the cooling agent is introduced to the conveying gas for pneumatic transportation of the fine-particle, pulverulent solid substances, before the conveying gas comes into contact with the solid substances.
- 10 18. The method as claimed in Claim 1, characterised in that the cooling agent used for direct cooling contains less than 0.0001 parts by mass of water and in this way the dew point of the gas phase containing solids is lowered.
- 15 19. The method as claimed in Claim 1, characterised in that the fine-particle, pulverulent solid substances are filled in paper bags or plastic bags on completion of cooling.
- 20 20. The method as claimed in Claim 1, characterised in that the fine-particle, pulverulent solid substances are filled in silo vehicles on completion of cooling.
21. Fine-particle, pulverulent solid substances in bulk material packings and produced as claimed in Claim 1, characterised in that the composition of the gas phase between the pigment particles contains less than 20% by weight oxygen, preferably less than 15% by weight oxygen.
- 25 22. Fine-particle, pulverulent solid substances in bulk material packings and produced as claimed in Claim 1, characterised in that the composition of the gas phase between the pigment particles contains a mass ratio of nitrogen to oxygen of more than 4, preferably of more than 5.7.
- 30 23. A device for direct cooling of fine-particle, pulverulent solid substances as claimed in Claim 1, characterised in that this device consists of a reservoir for low-boiling, condensed gases, an insulated connection pipe between the reservoir and a supply pipe for pneumatic conveying of the fine-particle, pulverulent solid substances, a nozzle for introducing the low-boiling, condensed gases to the
- 35 supply pipe and a control and regulating device.