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(54) **PROCESS FOR PREPARING GRANULAR UREA PRODUCT**

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(57) **ABSTRACT**

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To effectively prepare granular urea that constantly has a moisture concentration of 0.3 wt % or less in the product and has a large grain load strength to be hardly crashed. In a granulating process for preparing granular urea from an aqueous urea solution using a fluidized bed method or a fluidized, spouted bed method, an aqueous urea solution having an urea concentration of 94-98.5 wt % is used, and an operation temperature of the fluidized bed is controlled in a range of 110-120° C. to accelerate drying of a granulated product, thereby giving a granular urea product having moisture of 0.3 wt % or less in the product.

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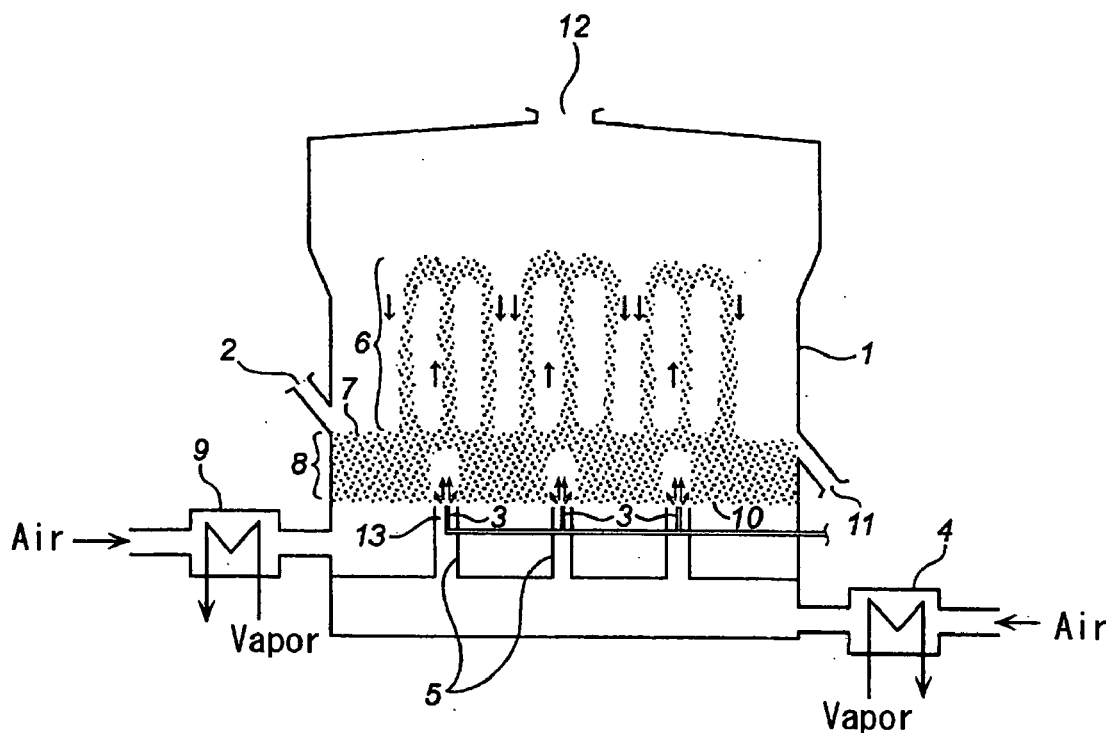


FIG. 1

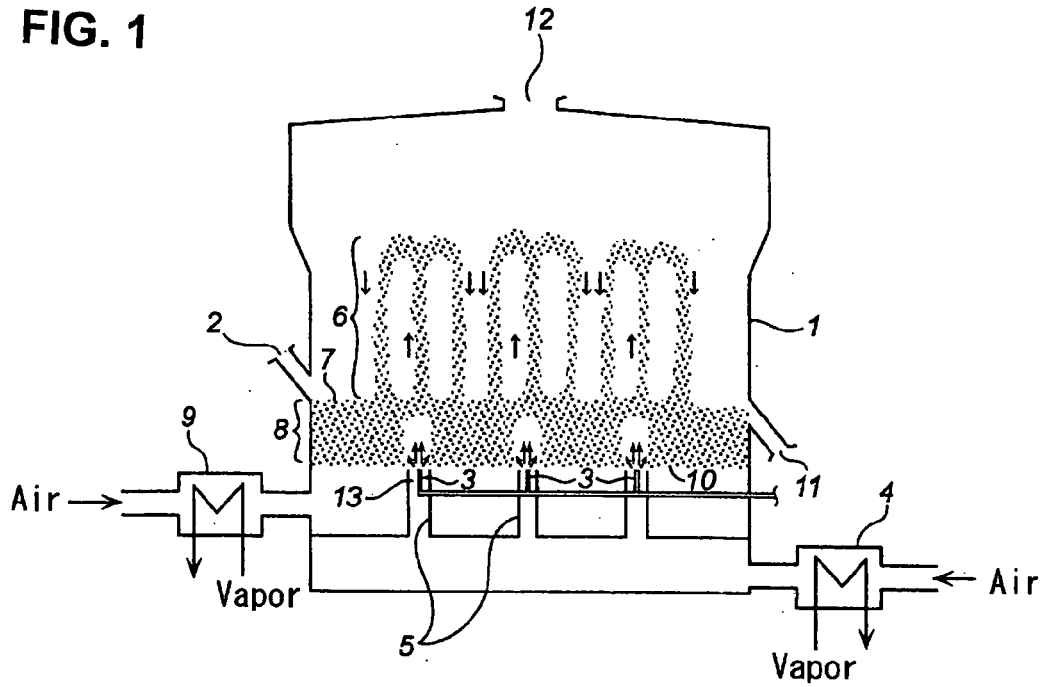
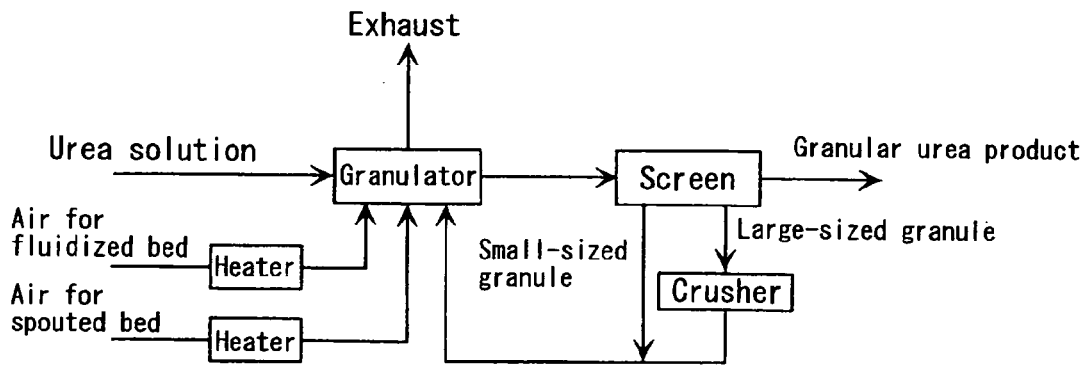


FIG. 2



PROCESS FOR PREPARING GRANULAR UREA PRODUCT

TECHNICAL FIELD

[0001] The present invention relates to a process for preparing granular urea from an aqueous urea solution, wherein it relates to a process for preparing a product having a high product strength by sufficiently lowering moisture in a granular urea product.

RELATED ARTS

[0002] With regard to a process for preparing relatively large-sized granular urea, in old times, there were a method using a drum and a method using a dish type granulator. However, these methods had such drawbacks that: in granulating urea, solidification heat of urea is released and a recycle operation is required, in which granulated urea is returned to a granulator after being cooled so that a granulator be operated under an interior temperature condition appropriate to granulation; the recycle requires a large facility; one granulator has limitations in production quantity; plural granulators must be installed for recent industrial scale; and urea sprayed into a granulator is a molten urea with the same moisture content as that in a product.

[0003] Therefore, recently, granulating processes employing a fluidized bed or a fluidized, spouted bed become mainstream, and various techniques have been proposed in these granulating processes.

[0004] JP-B 4-63729 proposes a method for preparing granular urea by scattering a spouted bed in a fluidized bed and supplying coating material for grains as fine drops to process grains in the bed. Specifically, it proposes a urea granulating method that defines thickness of a fluidized bed, space for grains to be processed to jump up and fall down, spraying angle for a coating material, spouting velocity and flow volume of air for forming a spouted bed, an arranging method of the spouted bed, and the like. In this urea granulating method, since solidification heat of urea is removed by an air flow forming a spouted, fluidized bed, granulated and cooled granular urea is not required to be returned again to a granulator, whereby a granulating facility is simplified. In addition, there is such a merit that the granulator makes production possible by one plant irrespective of production scales.

[0005] Further, JP-A 10-216499 proposes a process that further improves the fluidized, spouted bed method proposed in JP-B 4-63729. Specifically, it discloses that, when carrying out granulation by using a granulator constituted of a granulation section, an air-supplying pipe for spouting air to the granulation section, a nozzle for ejecting a stock liquid arranged at the center portion of the air-supplying pipe outlet, a bottom floor in which a fluid flows, and an upside supply port for supplying air for fluidization to the bottom floor, and ejecting a molten stock liquid or a stock liquid in slurry from the nozzle for ejecting a stock liquid to the granulation section, by determining (a) the distance between the air-supplying pipes to be 100 to 1000 mm, (b) the ejection angle of the nozzle for ejecting a stock liquid to be 30 to 60 degrees, (c) the ejection volume of the nozzle for ejecting a stock liquid to be 0.5 to 1.2 liters per 1 m³ of air volume at the air-supplying pipe outlet, (d) the linear velocity at the air-supplying pipe outlet to be 1.2 to 3.5 times the

terminal velocity of an average granule, and (e) the linear velocity of air supplied to the bottom floor in which granulated products are fluidized to be 1.2 to 3.5 times the minimum fluidizing velocity, a stabilized fluidized bed and a spouted bed are formed, a product containing no deformed product can be prepared, and an air consumption to be supplied to a granulator including the bed portion and air-supplying pipe can be reduced to achieve a significant energy saving effect.

[0006] In JP-B 56-47181, there is proposed such process for achieving granulation as spraying an aqueous urea solution, using a nozzle that sprays it together with air, on urea nuclei fluidized in a form of fine droplet, that is a droplet having an average diameter of 20 to 120 μm , and giving such a temperature that evaporates water from the solution sprayed on the nuclei and crystallizes urea on the nuclei to form a granulated product having a desired dimension. It is described that, in this process, the created granules are subjected to drying treatment with air at 100 to 150° C. to keep the temperature of the granule at 70 to 90° C., in order to remove moisture on the surface.

[0007] In Nitrogen & Methanol November-December 2004 pp 37-43, a urea granulation technique of Stamicarbon Company is presented. The process is a granulating process characterized by a Film spraying nozzle. There are such descriptions that urea as a stock to be sent to a granulator is made into a 98.5 wt % aqueous urea solution, and operation is carried out while adjusting the temperature of the fluidized bed at about 105° C. Moisture of the granular urea prepared by the method is described as 0.3 wt % or less without specific value. Further, there is no description about product quality when a urea liquid having concentration of 98.5 wt % or less is used as a stock.

[0008] Generally, in order to further concentrate an aqueous urea solution of 95 wt %, addition of a relevant concentrating system, a condenser for evaporating water and a vacuum generator is necessary, which requires a heavy investment. Therefore, use of a urea solution with a lower urea concentration as a stock is better. Further, in a granulator, heat generates when urea solidifies, which must be removed. The removing of the heat is often carried out with air, and in a generally used granulator employing a fluidized bed, and urea solution with a lower urea concentration can remove the heat through evaporation of water to make it possible to reduce a volume of air necessary for a fluidized bed.

DISCLOSURE OF THE INVENTION

[0009] The background art is a process and a facility for preparing granular urea from a molten urea as a stock by using a granulator constituted of a fluidized, spouted bed. There is no specific citation about moisture concentration in the product prepared there, and in JP-A 10-216499, although there is such description about a role of air for a jet flow of an air-supplying pipe that it solidifies a molten product to granulate it and, at the same time, removes moisture contained in the molten product to dry, and that drying is carried out thus in a jet flow section, there is no citation about the effect. Generally, with regard to the product quality of a granular urea, important items are the total nitrogen content, biuret as an impurity and moisture. Generally, the moisture of 0.3 wt % or less is a standard value and a designed value

of a urea product at the shipment thereof. Products with moisture of 0.3 wt % or more are sometimes marketed, actually. However, a high moisture in urea lowers product hardness to result in heavy abrasion in instruments that handle the product and increase in generation of urea dust, or, on storage in a warehouse, the moisture evaporates and comes into contact with a cold ambient air to wet the surface of the urea product, which leads to the agglomerate of urea grains with each other to form a large agglomerate, whereby it may become in such condition that does not allow it to be taken out as a finished product. Thus, a moisture as low as possible is desirable and, in particular, when handling a granular urea product granulated by utilizing a fluidized bed, that of 0.3 wt % or less is preferred.

[0010] Since a granulator utilizing a fluidized bed has a drying effect, an aqueous urea solution of 94-95 wt % or more is often used as a stock. However, some granulator may result in moisture of 0.3 wt % or more in a prepared product when starting from a stock of 94-95 wt %. On this occasion, concentration of a stock urea solution must be increased to 98-99.7 wt %, instead of a 94-95 wt % aqueous urea solution, to assist the drying effect so that moisture in the product is 0.3 wt % or less. However, although heat generated when urea agglomerates is removed by air sent to a granulator and evaporation heat resulting from evaporation of water in an aqueous urea solution, when the concentration of an aqueous urea solution is increased, evaporation heat of water is decreased to increase the volume of air for cooling and capacity of a blower, which is not preferred because power consumption necessary for granulation is increased and production cost is pushed up.

[0011] Some granulators employ such a process that an aqueous urea solution is sprayed while introducing air of a high temperature into a nozzle for spraying to carry out granulation while drying fine aqueous urea solution particles. In this process, however, it is required to increase air pressure to a pressure necessary for spraying an aqueous urea solution, in general to 1 to 2 bar, which requires a considerable motive energy for increasing air pressure and is not economical.

[0012] Solidification heat of urea is removed by air in a fluidized bed, however, since agglomeration temperature of urea is about 132° C., a low temperature in a granulator results in formation of granular urea agglomerates having a so-called confetto-like shape formed by sticking of urea grains while using a sprayed urea as a binder because agglomeration on the urea grain surface is fast, which is not preferable. Accordingly, a granulator is often operated at a low temperature in a range that does not cause the above-described problem, that is, at 95-105°, because urea grains leaving the granulator are cooled. On the other hand, with regard to operation under elevated temperatures, since there are concerns about miscarrying in urea granulation in a granulator at a temperature too close to an agglomeration temperature, temperature of a fluidized bed is not elevated beyond the above-described value.

BRIEF DESCRIPTION OF THE DRAWINGS

[0013] FIG. 1 is a cross-sectional view of an embodiment of a granulator of the fluidized, spouted bed method as one example of the present invention.

[0014] FIG. 2 is a general flow for preparing granular urea by using a granulator.

[0015] Further, in the diagram, the code 1 means a granulator, 2 means a seed grain-supplying port, 3 means a nozzle, 4 means an air heater for a spouted bed, 5 means an air-supplying pipe, 6 means a space, 7 means an upper portion of a fluidized bed, 8 means a fluidized bed, 9 means an air heater for a fluidized bed, 10 means a bottom floor, 11 means a granule discharge port, 12 means an exhaust port and 13 means an upper portion of an air-supplying pipe.

[0016] The present invention provides a process for decreasing moisture in a product to 0.3 wt % or less when an aqueous urea solution is used as a stock solution in a granulator utilizing a fluidized bed.

[0017] The present invention has been achieved by following means.

[0018] The present invention is a process for preparing granular urea product having moisture of 0.3 wt % or less, which comprises the steps of feeding an aqueous urea solution having an urea concentration of 94-98.5 wt % in a granulator employing a fluidized bed method or a fluidized or spouted bed method, and granulating it at an operation temperature of the fluidized bed of 110-120° C., while accelerating drying of a granulated product.

[0019] In other words, the invention is a granulating process for preparing granular urea product from an aqueous urea solution by using a granulator employing a fluidized bed method or a fluidized, spouted bed method, wherein an aqueous urea solution having an urea concentration of 94-98.5 wt % is used and an operation temperature of the fluidized bed is controlled in a range of 110-120° C. to accelerate drying of a granulated product, thereby giving a granular urea product having moisture of 0.3 wt % or less in the product.

[0020] More specifically, it is a process, in a granulating process using a granulator constituted of an air-supplying pipe for ejecting air to a fluidized bed fluidizing a granulated product, a nozzle for ejecting a stock urea solution arranged at the center portion of the pipe, and a porous plate for supplying fluidizing air to the fluidized bed, and carrying out granulation by ejecting the urea solution from the nozzle for ejecting a stock urea solution to a granulation section, wherein an aqueous urea solution with a urea concentration of 94-98.5 wt % is used and an operation temperature of the fluidized bed is controlled in a range of 110-120° C. to accelerate drying of the granulated product to give a granular urea product with moisture of 0.3 wt % or less.

[0021] Further, the process is also effective in a process for carrying out granulation using a granulator in which a stock urea solution is sprayed to a fluidized bed that is fluidizing a granulated product while introducing compressed air into a nozzle for ejecting the stock urea solution, thereby solidifying and granulating urea on the granulate in the fluidized bed. That is, although a moisture concentration in a product was affected by volume of a compressed air and spraying performance of a spray, by operating a fluidized bed in a temperature range of 110-120° C., it becomes possible to stably give a product with moisture of 0.3 wt % or less without the above-described affection.

[0022] In addition, the present inventors discovered that a slight increase in temperature of a fluidized bed has no affection on urea granulation, and that an elevated temperature makes it possible to manufacture a granulated product

with a lowered moisture in the urea product and a high hardness. However, the drying effect in the fluidized bed is of no effect when the temperature of the fluidized bed becomes less than 110° C., because moisture of the granule at the granulator outlet exceeds 0.3 wt %. In a range of up to 120° C., a higher fluidized bed temperature has a larger drying effect, but above 120° C., an urea solution drop adhering to the urea grain surface does not instantly solidify on the surface and requires some time period to solidify. Since a granular urea that has solidified slowly has a low strength and wears in a solid handling instrument to generate powder, whereby continuous operation of a granulating facility becomes impossible. Thus, the temperature must be kept at 120° C. or less.

[0023] According to the present invention, it is possible to prepare a granular urea constantly having a moisture concentration of 0.3 wt % or less in the product. Since the moisture is low, it is a granular urea having a large granule load strength and being nonbreakable, it hardly breaks in an instrument and warehouse handling the product and generates a little dust.

[0024] Hereinafter, as an example of embodiments of the present invention, an example according to a fluidized, spouted bed granulator (FIG. 1) in which a spouted bed is arranged to a fluidized bed is described. But, needless to say, the present invention can be applied to a granulator employing a fluidized bed.

[0025] FIG. 1 is a cross-sectional view of an example of a fluidized, spouted bed granulator.

[0026] To granulator 1, a seed grain for urea to be granulated is supplied from supply port 2. A urea solution is sprayed to the granulator 1 from spray nozzle 3 located at upside outlet 13 of air-supplying pipe 5 for a spouted bed. The seed grain grows in the grain diameter by receiving the spray of urea solution within the granulator 1, which upflies to space 6 by a spouting air flow having been heated by air heater 4 for the spouted bed, introduced to a lower portion of the granulator, and split by plural air-supplying pipes 5, and then falls again on upper portion 7 of the fluidized bed. Through air heater 9 for the fluidized bed, air for the fluidized bed is supplied to the granulator 1, granular urea having grown on bottom floor 10 is in a fluidized state in fluidized bed section 8, and the granular urea is fluidized so as to fill the whole space above the nozzle 3. Such movement is repeated, the granular urea gradually grows, and, finally, the completely granulated granular urea is discharged from vent 11.

[0027] Urea is granulated by using the granulator 1, wherein, as described above, the granulation is practiced in the granulator by cooling and solidifying of a urea solution stuck to a seed grain, and the cooling is practiced with air for the spouted bed and air for the fluidized bed. The temperature of the fluidized bed changes depending on production volume, air volume, urea solution concentration and temperature of air for the fluidized bed. In particular, since atmospheric air is used as the air for fluidized bed, the temperature thereof is affected by atmospheric air temperature. Further, amount of evaporation heat of water contained in a urea solution changes depending on concentration of the solution, and a lower concentration requires more heat for evaporation to lower the temperature of the fluidized bed. Therefore, the air for the fluidized bed is heated with the air

heater 9 for the fluidized bed and introduced into the granulator 1 to stabilize the temperature of fluidized bed section 8 at 110-120° C. irrespective of ambient temperature and concentration of a stock aqueous urea solution. As the result, moisture content contained in the product can be maintained at 0.3 wt % or less.

[0028] In a granulating process characterized by using a Film spraying nozzle, the process of the present invention can decrease moisture content in the product by increasing the fluidized bed temperature, even when stock urea concentration sent to the granulator is 98.5 wt % or less.

EXAMPLES

[0029] Next, the present invention shall be described in further detail based on Examples. However, needless to say, the present invention is not limited only to these examples.

Example 1

[0030] By using a fluidized, spouted bed granulator, 41.7 ton/h of granular urea is produced. The flow is shown in FIG. 2.

[0031] 48 air-supplying pipes were prepared, and, at the upside of respective air-supplying pipes, a spray nozzle for spraying a stock urea solution was disposed. From the supply port 2, a seed grain was sent to start operation, and 0.943 ton/h of a 95 wt % aqueous urea solution was sprayed from each of the spray nozzle, which corresponded to 45.26 ton/h summed for the granulator, to prepare granular urea. At that time, 95,800 Nm³/h of air at 35° C. was sent to the fluidized bed so that the air had a velocity of 2 m/s. On the other hand, the total 25,700 Nm³ of air having been heated up to 130° C. by the air heater 4 for the spouted bed to the 48 air-supplying pipes. The granular urea from the granulator discharge port 11 was cooled down to 90° C., which was then classified with a screen to give granular urea having 2-4 mm product size as a urea product, followed by cooling to a product temperature of 50° C. to be sent to a warehouse. Among granules lying outside the product size, larger ones were crashed with a crusher after cooling down to 70° C., and smaller ones were returned directly to the granulator as the seed grain. In the operation, temperature of the fluidized bed 8 was 104° C. As the result of the operation, 68.85 ton/h of granular urea was discharged at 105° C. from the granulator discharge port 11, which was passed through a screen to give 41.7 ton/h of 2-4 mm urea product to be sent to a warehouse. On the other hand, 27.15 ton/h of granular urea separated by the screen was returned to the granulator as the seed grain, as mentioned above.

[0032] Difference of urea amount between the sum of 27.15 ton/h returned to the granulator as the seed grain and urea in the stock aqueous urea solution, and the granular urea amount discharged from the granulator, was discharged as dust from an upside air discharge port of the granulator 1 together with the air for fluidized bed, the air for spouted bed and evaporating water in the urea granulation. The discharged gas containing the urea dust was washed in an exhaust gas-cleaning tower with a circulating liquid. The urea was collected as an aqueous urea solution and returned to a urea factory. Moisture contained in the product was in a range of 0.35-0.4 wt %, and load strength of granular urea with a size of 3 mm in diameter was 2.5-3 kg.

[0033] Consequently, in the above-described operation state, the fluidized bed temperature was gradually increased by heating the air for fluidized bed. When it exceeded 110° C., moisture in the product tended to fall to below 0.3 wt %. By further heating the air for fluidized bed up to 55° C., the fluidized bed temperature was raised up to 115° C. As the result, moisture in the obtained granular urea was lowered to 0.2-0.25 wt %, and load strength of granular urea with a size of 3 mm in diameter also increased to 3.5-4 kg.

[0034] Further, when the air for fluidized bed was heated to raise the fluidized bed temperature up to 121° C., clogging at the screen by urea powder and plugging in a shoot for solid handling occurred, which did not allow the facility to continue the operation. It is thought that, since the fluidized bed temperature was high, the sprayed aqueous urea solution solidified slowly on the grain surface and since the thus prepared granular urea had a low strength under a rather high temperature condition, wear thereof occurred in the solid handling instrument to generate powders.

Example 2

[0035] In the same operation state as in Example 1, a urea solution of 98 wt % in concentration was used to practice granulation. At that time, temperature of the air for fluidized bed was 38° C. Since the urea solution contained a small amount of water, the temperature of fluidized bed increased and was 115° C. during operation even when temperature of the air for fluidized bed was 38° C. Moisture contained in a product at that time lowered down to 0.2-0.25 wt %. Load strength of granular urea with 3 mm in diameter was 3.5-4 kg. Then, since ambient air temperature lowered to 25° C., the fluidized bed temperature lowered down to 106° C. Moisture in a product at that time increased up to 0.35-0.4 wt %. Accordingly, temperature of the air for fluidized bed was raised up to 38° C. to result in the fluidized bed temperature of 115° C. for the purpose of making the moisture be 0.3 wt % or less, whereby moisture in the product decreased down to 0.2-0.25 wt % to produce granular urea having a diameter of 3 mm and load strength of 3.5-4 kg.

Example 3

[0036] In the same flow as in Example 1, a granulator, which practices spraying to a fluidized bed fluidizing a granulated product by introducing compressed air into a nozzle for spraying a stock urea solution and solidifies urea on the granulated product in the fluidized bed, was used.

[0037] 144 of two-fluid spray nozzles for spraying a urea solution by introducing compressed air into a nozzle for spraying a stock urea solution were disposed. A seed grain was sent from the supply port 2 to start operation, and 0.314 ton/h of a 95 wt % aqueous urea solution was sprayed from each spray nozzle, that is, 45.22 ton/h summed for the granulator, to prepare granular urea. To the 144 of spray nozzles, 23,040 Nm³ in total of air having been heated up to 130° C. by the air heater 4 were sent. At that time, 95,800 Nm³/h of air at 35° C. was sent to the fluidized bed so as to have an air velocity of 2 m/s. Granular urea from the granulator discharge port 11 was cooled down to 90° C., from which granular urea having a product size of 2-4 mm was then classified with a screen as an urea product, which was cooled down to a product temperature of 50° C. and

then sent to a warehouse. Among granules lying outside the product size, larger ones were crashed with a crusher after cooling down to 70° C., and smaller ones were returned directly to the granulator as the seed grain. In the operation, temperature of the fluidized bed 8 was 104° C. As the result of the operation, 68.79 ton/h of granular urea was discharged at 105° C. from the granulator discharge port 11, which was passed through a screen to give 41.6 ton/h of 2-4 mm urea product to be sent to a warehouse. On the other hand, 27.19 ton/h of granular urea separated by the screen was returned to the granulator as the seed grain, as mentioned above.

[0038] Difference of urea amount between the sum of 27.13 ton/h returned to the granulator as the seed grain and urea in the stock aqueous urea solution, and the granular urea amount discharged from the granulator, was discharged as dust from an upside air discharge port of the granulator 1 with the air for fluidized bed, the air for two-fluid spray nozzle and evaporating water in the urea granulation. The discharged gas containing the urea dust was washed in an exhaust gas-cleaning tower with a circulating liquid. The urea was collected as an aqueous urea solution and returned to a urea factory. Moisture contained in the product was in a range of 0.3-0.35 wt %, and load strength of granular urea with a size of 3 mm in diameter was 3-3.5 kg.

[0039] Consequently, in the above-described operation state, the fluidized bed temperature was gradually increased by heating the air for fluidized bed. When it exceeded 110° C., moisture in the product tended to fall to below 0.3 wt %. By further heating the air for fluidized, the fluidized bed temperature was raised up to 115° C. As the result, moisture in the obtained granular urea was lowered to 0.2-0.25 wt %, and load strength of granular urea with a size of 3 mm in diameter also increased to 3.5-4 kg.

[0040] Further, when the air for fluidized bed was heated to raise the fluidized bed temperature up to 121° C., the same state as in Example 1 was resulted in and operation was stopped.

Example 4

[0041] The two-fluid nozzle in Example 3 was replaced by a two-fluid nozzle that sprays a urea solution in a liquid film of inverted cone shape (Hollow Cone), and operation was practiced under the same conditions as in Example 3. Moisture contained in a product prepared by the process was in a range of 0.35-0.4 wt %, and load strength of granular urea with a size of 3 mm in diameter was 2.5-3 kg. In the above-described operation state, the fluidized bed temperature was gradually increased by heating the air for fluidized bed. When it exceeded 110° C., moisture in the product was measured to give a value of below 0.3 wt %. By further heating the air for fluidized bed, the fluidized bed temperature was raised up to 115° C. The same result as in Example 1 was obtained, that is, moisture of the obtained granular urea lowered to 0.2-0.25 wt %, and load strength of granular urea with a size of 3 mm in diameter also increased to 3.5-4 kg.

[0042] Further, when the air for fluidized bed was heated to raise the fluidized bed temperature up to 121° C., the same state as in Example 1 was resulted in and operation was stopped.

1. A process for preparing granular urea product having moisture of 0.3 wt % or less, which comprises the steps of feeding an aqueous urea solution having an urea concentration of 94-98.5 wt % in a granulator employing a fluidized bed method or a fluidized or spouted bed method, and granulating it at an operation temperature of the fluidized bed of 110-120° C., while accelerating drying of a granulated product.

2. The granulating process according to claim 1, wherein granulation is carried out using a granulator comprising an air-supplying pipe for ejecting air to a fluidized bed fluidizing a granulated product, a nozzle for ejecting a stock urea

solution arranged at the center of the pipe and a porous plate for supplying air for fluidization to the fluidized bed, and ejecting an urea solution from the nozzle for ejecting a stock urea solution.

3. The granulating process according to claim 1, wherein granulation is carried out using a granulator in which a stock urea solution is sprayed to a fluidized bed that is fluidizing a granulated product while introducing compressed air into a nozzle for ejecting the stock urea solution, thereby solidifying and granulating urea on the granulated product in the fluidized bed.

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