(54) Method for preparation of complex fertilizers

The [present] invention relates to a method for the preparation of mineral fertilizers, in particular, of chlorine-free nitrogenous potassium fertilizers. The present method for the preparation of complex fertilizers constitutes a process for preparing ammonium nitrate by the conversion of calcium nitrate with ammonium carbonate, wherein 4-21% of the ammonium carbonate is replaced by potassium carbonate and the mixing conversion yields a solution with total a salt mass fraction of 60 ± 5%, which contains a potassium and ammonium nitrate mass ratio of (5-25):(95-75), respectively. The obtained solution is either used as a liquid complex fertilizer or the water is evaporated and [the residue is] granulated by prilling. The technical effect is a simplification of the method for preparing a nitrogenous potassium fertilizer that contains potassium and ammonium nitrate and an increase in its cost effectiveness.
[1 claim and] 2 dependent claims.

Specification

The present invention relates to a method for the preparation of mineral fertilizers, in particular, to chlorine-free nitrogenous potassium fertilizers that contain nitrogen and potassium in a water-soluble form that can be used in the production of complex fertilizers.

A method is known for the preparation of nitrogenous potassium granulated fertilizers [RU patent № 2154620, C 05 C 1/02, 14.01, 2000], which comprises mixing ammonium nitrate and potassium chloride followed by granulation of the mixture obtained in a drum granulator; in this case, ammonium nitrate is used in the form of fusion cake. The nitrogenous potassium fertilizer prepared in accordance with said method contains 12-25% of nitrogen and 16.6-40% of potassium calculated as potassium oxide.

The disadvantages of the above method are as follows:

- reduction of the agrochemical value of the fertilizer due to the presence of chloride ion in the finished product;
- technical and equipment complexities in implementing the process of mixing ammonium nitrate in the form of fusion cake with potassium chloride;
- increased corrosion of the facilities and equipment because of the introduction of chloride ions into the N:K fertilizer preparation;
- an increasing hazards in the equipment operation associated with mixing the fusion cake with potassium chloride due to the strong catalytic action of chloride ions on the ammonium nitrate thermal decomposition process (Reference book for nitrogen investigators./ Edited by Ye. Ya. Melnikov, p. 157, Khimiya, 1987).

The most similar in the technical essence and the result obtained to the method offered is the method for the preparation of complex N:K fertilizer [RU patent № 2182144, C 05 G 1/00, 09.01.2000].

This method includes the preparation of ammonium nitrate from ammonia and nitric acid, the addition at the neutralization stage of potassium nitrate in ammonium nitrate in the form of a 30-70% solution in an amount based on 2-20 mass% of the ammonium nitrate, the evaporation of the mixed solution and its granulation.

The nitrogenous potassium fertilizer contains 34.2-30.4 mass% of nitrogen and 0.9-9.3 mass% of potassium calculated as potassium oxide.

The disadvantages of the method are as follows:

- the necessity of using the most expensive potassium raw material – potassium nitrate, which increases the cost of the N:K fertilizer production;
- the difficulty associated with the regulation of the composition and the salt ratio in the finished product, when potassium nitrate is introduced to the neutralizer at the [stage of] ammonia neutralization with nitric acid;
- the necessity of additional equipment for the preparation of potassium nitrate solution and its dosing into the neutralization vessel;
- increased equipment loading associated with the stage of evaporation of the mixed solution of ammonium and potassium nitrate to achieve the fusion cake form, since potassium nitrate is used in the form of aqueous solution.

The objective of the present invention is the development of a simplified and economically feasible method for the preparation of a mixed solution containing potassium and ammonium nitrate in a specified ratio followed by the processing of said solution into complex liquid and chlorine-free, granulated complex fertilizers, which contain nitrogen and potassium in a water-soluble form.

The objective is realized in accordance with the offered method through the preparation of a mixed solution containing potassium and ammonium nitrate derived from calcium nitrate, in particular, the calcium nitrate obtained in the NPK fertilizer production. The calcium nitrate obtained in the NPK fertilizers production contains impurities, which restrict its direct use; therefore, calcium nitrate is processed by the conversion with ammonium carbonate into ammonium nitrate and calcium carbonate (chalk).

To realize the objective according to the present invention, a portion of ammonium carbonate is replaced with potassium carbonate in the process of the calcium nitrate conversion, which results in obtaining a mixed solution that contains potassium and ammonium nitrate.

The ratio of potassium and ammonium nitrate in the mixed solution is determined based on the ratio of potassium and ammonium carbonate that are fed into the conversion reactor. The consumption

of potassium carbonate in the conversion process is regulated depending on the requirements for the composition of the finished product. Complete calcium nitrate conversion is provided by adding ammonium carbonate; the process is monitored by the excess of carbon dioxide in the calcium carbonate slurry.

The conversion reactor is charged with potassium carbonate either in the form of a sesquihydrate or in the form of an aqueous solution with a potassium carbonate mass fraction of 50 ± 5%. Ammonium carbonate is fed into the conversion reactor in the form of a solution that contains an ammonium carbonate mass fraction of 30 ± 5%, prepared via absorption of ammonia and carbon dioxide by a mixed solution of potassium and ammonium nitrate in an absorption column.

For the granulation of the complex fertilizer prepared from the fusion cake by using the equipment for the pure ammonium nitrate production, the mass ratio of potassium and ammonium nitrate in the mixed solution is regulated within the range of (5-25):(95-75). The optimal composition of the ammonium and potassium nitrate mixture for the granulation by prilling is chosen based on data for the fusion cake crystallization temperature, which is given in an article by L. I. Olefir, A. I. Mishchenko “Investigation of physicochemical properties of complex chlorine-free NK-type fertilizers” [Khimicheskaya promyshlennost’ (Chemical industry), 1983, № 3, p. 162].

Solutions with the above salt ratio are prepared in the calcium nitrate conversion process by replacing 4-21% of the ammonium carbonate with the dry substance equivalent of potassium carbonate.

After the converted calcium carbonate has been separated and rinsed, and the mixed solution has been purified from particulate matter, the mass fraction of potassium and ammonium nitrate is in the solution 60 ± 5%. The prepared mixed solution can be used as a liquid complex fertilizer for drip irrigation.

In order to obtain a granulated complex fertilizer, the mixed solution that contains a 60 ± 5% mass fraction of potassium and ammonium nitrate and a salt ratio of (5-25):(95-75) is evaporated to a concentrated solution, in which the of the total salt mass fraction is 90 ± 3%.

The evaporation of the mixed solution is performed with the help of technology for ammonium nitrate preparation used in the production of NPK fertilizer, which is followed by the complete evaporation of the concentrated solution and the granulation of the fusion cake by prilling by using the ammonium nitrate production equipment.

The technical effect obtained is as follows:

- the possibility of preparing mixed solutions that have a specified ratio of potassium and ammonium nitrate;
- the use of a less expensive potassium-containing raw material – potassium carbonate;

- the expansion of the application field for calcium nitrate, which is an intermediate product in NPK fertilizer production;
- the preparation of a fertilizer by usoffing the equipment for NPK fertilizer and ammonium nitrate production without changing the technology standards;
- maintaining a water and energy balance in the ammonium nitrate production owing to the high salt concentration in the solution that is fed into the evaporation step and the granulation.

The method offered for the preparation of a complex fertilizer is economically feasible and simple to implement. Testing of the offered method was carried out through the use of a calcium nitrate melt and an ammonium carbonate solution taken from the production of NPK fertilizer and chemical reagents. The preparation of the potassium and ammonium nitrate mixture with a specified salt ratio according to the offered invention is illustrated by an example.

Example.

A conversion reactor was continuously charged with a calcium nitrate melt at 107 g/min, ammonium carbonate solution at 119 g/min and potassium carbonate at 12.9 g/min. The mass fraction of calcium in the calcium nitrate melt was 14.9%, the mass fraction of nitric acid was 3.7%, and the mass fraction of phosphates was 0.26% calculated as P_2O_5; other impurities were not regulated. In the solution, the mass fraction of ammonium carbonate was 31.2% and the mass fraction of potassium carbonate was 50.8%.

The slurry obtained was filtered, the calcium carbonate precipitate was separated and rinsed with water, and was fed into the drying [step]. After the slurry was separated and particulate matter had been removed, the filtrate was a mixed solution of potassium and ammonium nitrate that exhibited a salt mass fraction of 59.6% and a mass ratio of potassium and ammonium nitrate 11.4:88.6, respectively.

The mixed solution of potassium and ammonium nitrate was evaporated to a concentrated solution that contained a total salt mass fraction of 90 ± 3%, which was further evaporated to give a fusion cake that contained a water mass fraction of 0.4%, and then granulated by prilling.

In the granulated fertilizer thus obtained, the content of nutrients (in mass%) was: N – 32.5, K_2O – 5.3.

Claims

1. A method for the preparation of complex fertilizers comprising a preparation of a mixed solution that contains potassium and ammonium nitrate, wherein a mixed solution, in which the ratio of potassium and ammonium nitrate is regulated within the range of (5-25):(95-75), is prepared in the process of the calcium nitrate conversion, in which 4-21% of ammonium carbonate is replaced with potassium carbonate; potassium carbonate is fed into the reactor for conversion either into the form of a

sesquihydrate or into the form of aqueous solution that contains potassium carbonate mass fraction of (50 ± 5)%; the precipitate of converted calcium carbonate is separated by filtration, and the obtained mixed solution is used either as a liquid complex fertilizer or is evaporated; the fusion cake obtained thereby is granulated by prilling, where the complex chlorine-free fertilizers thus prepared contain nitrogen and potassium in a water-soluble form.

2. The method according to Claim 1, wherein calcium nitrate is prepared in NPK fertilizer production.

3. The method according to Claim 1, wherein calcium nitrate is prepared by the decomposition of natural calcium carbonate with nitric acid.