Patented Nov. 14, 1933 1,935,528

UNITED STATES PATENT OFFICE

1,935,528

PROCESS FOR THE SOLIDIFICATION OF REACTION PRODUCTS OBTAINED BY THE ACTION OF NITRIC ACID ON PHOSPHATE OF LIME

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No Drawing. Application July 25, 1931, Serial No. 553,233, and in Switzerland and Germany August 2, 1930

8 Claims. (Cl. 71—7)

This invention relates to the solidification of reaction products as are obtained by the reaction of phosphorites with nitric acid.

This reaction process has recently almost completely replaced the process hitherto chiefly used for the preparation of phosphate manures in which phosphorites are treated with sulphuric acid, because the products yield considerably better mixed manures.

Nevertheless the nitric acid reaction process has a number of disadvantages which to a certain extent nullify the considerable advantages over the sulphuric acid process.

Both processes first yield semi-liquid hot masses which are only converted into actual manures capable of being scattered by a following preparation or working up operation.

While in the superphosphate process relatively loose easily dividable masses are obtained by simply allowing the reaction product to solidify, the solidified nitric acid reaction products are extremely hard and their further treatment involves lengthy breaking and grinding processes.

Furthermore the preparation of the reaction mass for grinding after the reaction also presents a series of disadvantages of its own.

These disadvantages are partly due to the fresh reaction masses liberating considerable quantities of nitric acid fumes and to the fact that the solidification takes place extremely slowly and is accompanied by the formation of very hard products. The liberation of large quantities of nitric fumes not only imposes a considerable burden on the operators involving complicated and expensive protective measures, but also causes considerable damage to masonry and iron constructions in the operating chamber or requires specially protected chambers. Owing to the slow cooling and solidification due to the liberation of heat of crystallization, large solidifying chambers are necessary, with which is coupled the further difficulty that owing to the slow cooling, with large productions the individual charges in the solidifying chambers weld together to form extended masses, which cool very slowly in the interior and after solidifying are in the form of extremely hard pieces of large surface which renders the work of reduction considerably more difficult.

The process according to the invention completely overcomes all the above mentioned difficulties in the working up of reaction products by the nitric acid process.

Essentially it is based upon the still warm and plastic mass being subjected to an intermixing and mechanical subdivision treatment with the admixture of an already cold, reduced material or reaction mass of the same kind. It has been found particularly advantageous to employ the added cold reaction mass in finely divided form, advantageously ground into powder.

The product of addition used is therefore termed "powder" hereinafter but no limitation to this grade of subdivision is to be understood.

The preparation or working up of the still pasty reaction product at about 60° C. with cold powder has not only the advantage of considerable acceleration of cooling by more rapid removal of the heat of reaction and crystallization, but in addition welding together into large hard masses is avoided. The solidification is improved by the presence of cold powder not only because more rapid removal of heat takes place, but also because the crystal fragments of the powder act as crystallization germs which accelerate the crystallization of the reaction masses.

It has further been found that the admixture or coating of the fresh products with the powder practically completely suppresses the liberation of nitric acid fumes, so that the masses while being dealt with under the process according to the invention can be openly exposed without occasioning difficulties to the operators and without harmful effects on the masonry and constructional work of the operating chambers and apparatus.

The following modes of procedure have been found particularly advantageous in carrying out the invention, but they can be varied as desired according to requirements. To work up the fresh semi-liquid raw materials, the reaction can be effected in a kneading apparatus and the masses coming therefrom divided into large lumps, can be coated with powder advantageously by thoroughly kneading them with the powder. A mixing apparatus or the like can be used for this purpose. It has been found particularly advantageous however to use a rotary drum for this purpose, such a drum being of any known kind. The fresh masses and the powder are introduced together and thorough coating and intimate mixture of the raw mass and the powder obtained and also discharge effected, by rotation of the drum at a suitable inclination or by suitably moved members inside the drum. In some cases a dividing, or dividing and kneading or mixing device can be provided inside the drum. Such a dividing action can be obtained for example by a rotating shaft carrying knives

inside the drum, the divided masses being at the same time fed out of the drum by suitable arrangement of the knives.

The lumps leaving the drum at least superficially cooled by the treatment with or incorporation of the powder and accordingly in part solidified can, since they no longer liberate harmful fumes, be simply openly exposed in suitable chambers for final hardening without special protective measures. The powder treatment has the further advantage that the separate lumps can no longer weld together. In this way a preliminary reduction is already effected of the still plastic mass, which is considerably easier than after complete solidification, apart from the fact that the solidifying period is considerably reduced.

The preliminary reduction of the still plastic mass can be combined if desired with the powder treatment. For example such quantities of the cold powder can be added to the mass that at the same time as it is divided rapid solidification and reduction to the final degree, for instance to grain size, is possible, all undesirable phenomena such as welding together of the particles being avoided.

The quantity of added powder can however be determined exactly according to the desired degree of division of the fresh mass, so that the quantity of powder just suffices to prevent for example large pieces from welding together through being coated. Finally the added quantity of powder can for example be determined according to the desired speed of cooling or other operating requirements.

The cooling action of the powder can also be increased by other cooling measures known per se, for example by indirect cooling of the mass by passing it through or treating it in cooled apparatus, or by blowing cold air or other gas direct upon or into the material.

The material coming from the treating apparatus, either immediately or after further cooling, can be separated according to the desired grain size by known means, for example by sieving or by wind sifting. It is usually found advantageous to return too fine and too coarse material to the fresh masses.

Any of the various known devices for similar purposes can be used for the work of sub-dividing the reaction masses in plastic condition, and for cooling by admixture of the powder, if desired with the aid of other cooling measures.

Some modes of carrying out the invention are described in the following examples.

Example 1

By treating phosphorite with nitric acid in a kneading apparatus, a plastic mass is produced which after its exit from the apparatus is fed into a treating drum of the kind above referred to together with the powder. This feed can be uniformly effected by means of a worm.

The material is submitted to a certain degree of sub-division in the drum by means of knives rotating inside said drum, but preferably leaves the drum in lumps of for example the size of the fist, which by the powder treatment are superficially fairly well cooled and solidified. This material after being left exposed to the air for a short period of several hours is ready for further treatment in breakers and mills.

Example 2

After the reaction effected as in Example 1, the material, if desired after passing through a preliminary sub-dividing apparatus, is fed to a rotary drum of known kind at the upper end of which it meets with the powder. The reduction of the material and intermixing are further effected as in Example 1 but in such a manner that the material in a short time is solidified to a very considerable extent and leaves the drum for the most part with the proper size of grain. The grains are separated by wind sifting from the powder and from the part which has not been sufficiently reduced.

Example 3

The material, as in Example 2 is considerably cooled by the powder and at the same time reduced in a first rotary drum of the kind above referred to. It is cooled in a second similar rotary drum, but without special reducing devices, if desired with the aid of cold air; it is submitted to further sub-division in a third similar drum provided with reducing devices, until it reaches the desired grade of sub-division. In place of a cooling drum a conveyer band can be used upon which the already considerably reduced material is spread in a thin layer and in a short time is to a great extent solidified.

If it does not appear desirable to supply a sufficient excess of powder at the entry to the treating apparatus, any caking of still plastic grains at exit from the apparatus can be avoided by providing a further supply of dust at this point.

The invention has above been described as applied by way of example to the reaction of phosphorites with nitric acid. Its use is however not limited to this particular case of manure manufacture, but can be used whenever one stage in the manufacture of a manure leads to a pasty or semi-liquid material at high temperature, which for use must be in a finely divided form and where solidification of the material is necessary for the subdivision thereof.

What I claim is:—

1. A process for the solidification of the products of the reaction between phosphorites and nitric acid, said products being particularly adapted for use as a fertilizer, said process consisting of subjecting the plastic mass resulting from the said reaction, while still warm and during the course of its solidification to mechanical subdivision, while simultaneously admixing therewith a quantity of cold, dry and pulverized final product obtained in a previous operation.

2. A process for the solidification of the products of the reaction between phosphorites and nitric acid, said products being particularly adapted for use as fertilizer, said process consisting of subjecting the plastic mass resulting from the said reaction, while still warm and during the course of its solidification, to mechanical subdivision, while simultaneously admixing therewith a quantity of cold, dry and pulverized final product obtained in a previous operation, the quantity being determined by the degree of subdivision.

3. A process for the solidification of the products of the reaction between phosphorites and nitric acid, said products being particularly adapted for use as fertilizer, said process consisting of subjecting the plastic mass resulting from the said reaction, while still warm and during the course of its solidification, to mechanical subdivision, while admixing therewith an excess of cold and powdered final product from a previous operation, and then further admixing the result-

ant mixture with additional powdered material.

4. A process for the solidification of the products of the reaction between phosphorites and nitric acid, said products being particularly adapted for use as fertilizer, said process consisting of kneading the plastic mass resulting from the said reaction, while still warm, while admixing therewith a quantity of cold and dry final product obtained from a previous operation, and then subjecting the mass, during the course of its solidification, to mechanical subdivision.

5. A process for the solidification of the products of the reaction between phosphorites and nitric acid, said products being particularly adapted for use as fertilizer, said process consisting of subjecting the plastic mass resulting from the said reaction, while still warm and during the course of its solidification, to mechanical subdivision, while admixing therewith a quantity of cold, dry, subdivided final product from a previous operation, whereby the plastic mass is cooled, and supplementing the said cooling effect by subjecting the mass under treatment to an auxiliary external cooling.

6. The process set forth in claim 5, said auxiliary cooling treatment being effected by contacting said mass with cold gases.

7. A process for the solidification of the products of the reaction betwen phosphorites and nitric acid, said products being particularly adapted for use as fertilizer, said process consisting of subjecting the plastic mass resulting from the said reaction, while still warm and during the course of its solidification, to mechanical subdivision, while admixing therewith a quantity of cold, dry and subdivided final product from a previous operation, subjecting the mass to an auxiliary cooling treatment, and then admixing an additional quantity of said final product.

8. A process for the production of a fertilizer material comprising subjecting phosphorites to the action of nitric acid, whereby there is produced a plastic reaction mass containing a substantial proportion of hygroscopic and relatively slowly crystallizable calcium nitrate, subjecting the said reaction mass, while still warm and during the course of its solidification, to a mechanical subdivision treatment, while admixing therewith a quantity of final product from a previous operation.

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