

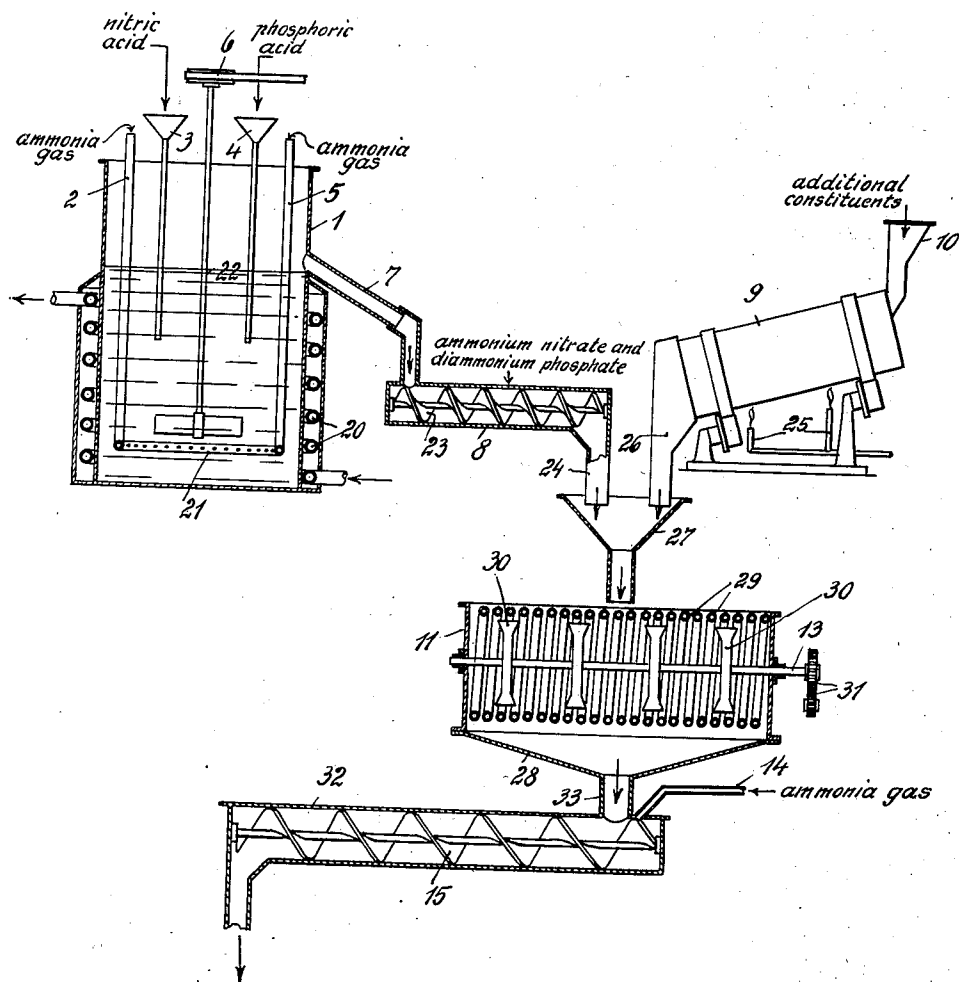
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PRODUCTION OF FERTILIZERS

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PRODUCTION OF FERTILIZERS

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Our invention relates to fertilizers and more particularly to means whereby fertilizers containing nitrogen and phosphorus alone or in combination with other fertilizing constituents can be produced in a simpler and more efficient manner than was hitherto possible.

As is well known to those skilled in the art, the production on a large scale of fertilizers containing, amongst other fertilizing constituents, also phosphate of ammonia, is connected with certain difficulties which result from the fact that solid diammonium phosphate $(\text{NH}_4)_2\text{HPO}_4$ could not be obtained directly. One of the principal difficulties encountered in the production of diammonium phosphate results from the fact that the production of highly concentrated phosphoric acid by acting with acids on mineral phosphates is not an easy matter since the mineral phosphates can only be decomposed for the recovery of free phosphoric acid by acting thereon with comparatively dilute acids, this being due to the necessity of effecting the decomposition in the presence of so much liquid that the suspension resulting in the reaction can readily be filtered. Apart therefrom the washing out of the products of decomposition, even if it is carried out in countercurrent, requires great quantities of water so that normally only a phosphoric acid of about 30% is obtained. In trying to concentrate this dilute phosphoric acid by evaporation other difficulties are encountered, which are created by the hydrofluoric acid admixed to the phosphoric acid and which is known to vigorously attack all vessels including ceramic vessels, when operating at high temperatures. All these reasons lead in many cases to the more expensive production of phosphoric acid by reduction of the phosphates and conversion of the phosphorus formed in this reaction in the gas phase into concentrated phosphoric acid.

We have now found that it is easily possible to produce solid phosphate of ammonia from dilute phosphoric acid by acting on mixtures of nitric acid and phosphoric acid with ammonia, whereby ammonium phosphate and ammonium nitrate are produced in one operation.

The new process according to this invention involves the further advantage that the ammonium phosphate thus obtained is mixed with ammonium nitrate which is one of the most valuable constituents of mixed fertilizers.

In the operation of our invention we prefer introducing simultaneously into the reaction vessel the ammonia and the mixture of phosphoric and nitric acids, utilizing the heat of reaction for concentrating the mixture. If a phosphoric acid

of low concentration, such as can readily be produced, is used in this operation, the heat of reaction may not be sufficient to completely evaporate the water introduced together with the acids, and in such case we prefer providing means for supplying heat from without either to the neutralizing vessel itself or to a separate vessel. In all cases care is taken to add the reagents in such proportions that the mixture is substantially neutral. We further prefer to operate in such manner that in spite of the evaporation of the water no solid ammonium nitrate separates out, but remains in the product of reaction in molten condition, serving as a means for suspending and transporting the solid phosphate which separates out.

Obviously the water contained in the phosphoric acid is here evaporated within the reaction mixture which has no corrosive action, and this water can be evaporated completely since, although the whole of the ammonium phosphate produced in the reaction is solid, the practically anhydrous molten ammonium nitrate acts as a means for suspending the ammonium phosphate, so that here the supply of heat from without, which is necessary in order to maintain the reaction mixture in a state of fluidity such that the danger of clogging of the pipes does not arise, does not encounter any difficulties.

During the reaction the mass should be vigorously stirred in order to maintain an intimate mixture. Preferably the mixture of molten ammonium nitrate and solid ammonium phosphate is withdrawn by means of an overflow or the like, in proportion as it is formed.

The principal advantage offered by this process lies in the production of a solid product being a mixture of ammonium phosphate and ammonium nitrate, which is formed directly on solidification of the reaction mixture.

If it is desired to produce mixed fertilizers we prefer adding the potassium salts, calcium salts, clayey or humus-like constituents to the solid mixture of ammonium nitrate and ammonium phosphate, the mixture of the additional constituents being heated so that during or after the mixture the ammonium nitrate is, at least partly, molten and the mass as a whole converted into a dough, which can then be caused to solidify by treating it on cooling cylinders, in cooling worm-conveyers or the like, the product being simultaneously or subsequently granulated.

If it is desired to obtain a predetermined acidity in the product, the mixing of the constituents may be effected in the presence of ammonia gas.

Example 1

In the drawing affixed to this specification and forming part thereof an apparatus adapted for use in the operation of our invention is illustrated diagrammatically by way of example in vertical section.

Referring to the drawing, 1 is the reaction vessel and 20 is a heating jacket surrounding the lower half of the vessel. 3 and 4 are funnels extending into the vessel and serving for introducing the nitric and phosphoric acids separately. 2 and 5 are pipes extending vertically into and in proximity of the bottom of the vessel, their bottom ends being connected with an annular perforated distributing member 21 through which ammonia gas is introduced. 22 is a stirrer driven by a belt 6. 7 is an overflow tube leading from the upper half of the vessel to the cylinder 8 surrounding a screw conveyer 23, 24 being a downwardly directed tube arranged at the outer end of the cylinder 8. 9 is a drum which can be heated from without as indicated by the burners 25 and 10 is a charging hopper serving for introducing the additional constituents of the mixed fertilizer, such as potassium salts, calcium salts, clay and the like. 26 is a discharge hopper. The discharge tube 24 and discharge hopper 26 are arranged above a hopper 27. 11 is a vessel arranged below the hopper 27 and provided with a sloping bottom 28. 29 is a heating coil arranged in the vessel 11, the windings of the coil being spaced in such manner that gaps 10 to 20 mms in width are formed between the windings of a coil. 13 is a horizontal shaft extending axially across the coil 29 and 30 are stirrer arms mounted on this shaft, which can be set rotating by driving means indicated at 31. 32 is the cylindrical casing of a worm conveyer 15 extending below the bottom 28 of the vessel 11 and connected therewith by a connecting tube 33. 14 is a pipe serving for introducing ammonia into the cylinder 32.

In the operation of this apparatus the nitric and phosphoric acids are introduced into the vessel 1 through the funnels 3 and 4, respectively, while ammonia gas is introduced through the pipes 2 and 5, issuing from the perforations of the distributor 21 and rising in the acid liquid, which is heated by the steam or hot liquid circulating in the jacket 20, to make up for any deficiency in heat beyond the heat of neutralization. The moist hot mixture of ammonium nitrate and diammonium phosphate forming in the reaction escapes from the vessel through the overflow tube 7 and is cooled in passing through the windings of the worm conveyer 8, dropping thereafter into the hopper 27 into which the additional constituents of the fertilizer to be produced, such as potassium or calcium salts, clay or the like, are distributed from the drum 9. The combined ammonium salts and other constituents are deposited on the heating coil 29 and being heated thereon are converted into a thickly fluid mass, the ammonia salts in the mixture being partly molten. The viscous mixture passing through the gaps between the windings of the heating coil 29 is caught and thoroughly worked through by the arms 30 of the stirring device operating within the coil, and escape from between the windings of the coil to drop onto the bottom 28 of the vessel and escape into the casing 32 of the conveyer worm 15, to be exposed therein, if desired, to the action of ammonia gas introduced through pipe 14. In the conveyer worm 15 the mixture is worked through and allowed to cool and is discharged at the outer end of the casing.

In practising our invention we may for instance proceed as follows:

Neutralization of the nitric and phosphoric acids is effected in the closed heated vessel 1 which may for instance have a capacity of 500 litres. First of all a molten mixture heated to 115° C. is filled into the vessel, consisting of 45% ammonium nitrate, 47% diammonium phosphate and 8% water. To this mixture is now added hourly a mixture of 47 kilograms=35.7 litres of a 50% nitric acid and 45.8 kilograms=34.0 litres of a 50% phosphoric acid. At the same time so much ammonia gas is introduced through the pipes 2, 5, that 13.7 kilograms NH₃ are absorbed per hour. During this operation the mixture in the vessel is kept constantly at 115° C. 64.8 kilograms of the molten mixture of ammonium nitrate and diammonium phosphate containing 7.3% water escape per hour through the overflow 7 in the form of a thin liquid and in this manner about 4 tons of a solid mixture can be recovered per day and cubicmetre capacity. The product is allowed to cool and solidify in the conveyer worm casing 8 and is thereafter mixed with 37 kilograms solid commercial potassium chloride containing 92% KCl. The mixture of the solid salts in passing through between the windings of the heating coil 29 is partly melted again and after having been worked through in the vessel 11 by the agitating device is allowed to drop into the conveyer worm below the coil where it is allowed to cool down. If desired ammonia gas may be introduced through the pipe 14, while the mixture is dried and allowed to solidify.

There are thus obtained about 100 kilograms of a mixed fertilizer containing about 16.5% N, 16.5% P₂O₅ and 21.5% K₂O.

Example 2

The neutralizing vessel having been filled with a molten mass heated to about 115° C., which is composed of 64% diammonium phosphate, 29% ammonium nitrate and 7% water, a mixture of 50.6 kilograms of a 30% phosphoric acid and 14.4 kilograms of a 50% nitric acid are introduced per hour, together with a quantity of ammonia gas sufficing to neutralize these acids. While the operation is proceeding, the temperature of the mixture in the vessel is maintained constant at about 115° C. About 6.6 kilograms ammonia are absorbed per hour and about 31.3 kilograms of a moist salt mixture containing ammonium nitrate and diammonium phosphate escape through the overflow, to be mixed, after cooling and solidification, with a mixture heated to about 100° C. of 58 kilograms potassium nitrate of 98% and 10 kilograms white clay free from carbonate. In passing through the coil 29 the mixture is heated to about 90° C. and is thereafter allowed to cool down and solidify in the conveyer worm 15. There are thus obtained per hour about 100 kilograms of a mixed fertilizer containing about 15% N, 11% P₂O₅, and 26.5% K₂O.

Example 3

To a molten mixture of 36% diammonium phosphate, 56% ammonium nitrate and 8% water are added hourly at 115° C. in the reaction vessel 71.6 kilograms of a 25% phosphoric acid, 53% of a 55% nitric acid and the quantity of 13.5 kilograms of the gas. Through the overflow there escape per hour about 64 kilograms of the mixture of moist ammonium nitrate and diammonium phosphate, which is now allowed to cool

and solidify, whereupon 35.6 kilograms potassium chloride of 98% and 10 kilograms dicalcium phosphate are added. The mixture in passing through the coil 29 is heated to 90-100° C., whereby the ammonium salts present in the mixture are partly melted. After cooling down in the conveyer worm below the heating coil about 108 kilograms of a mixed fertilizer are obtained per hour, which contains about 16.2% N, 12.0% P_2O_5 , and 20.4% K_2O .

Obviously the vessel 1 and the drum 9 may be heated in a different manner than here shown, and the means for mixing and cooling the intermediary and final products may be varied without departing from the spirit of our invention.

Apart from the apparatus here shown by way of example various changes may be made in the details disclosed in the foregoing specification without departing from the invention or sacrificing the advantages thereof.

We claim:—

1. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, allowing said mixture to cool down and solidify and adding to the solid mixture of ammonium salts another fertilizing agent under conditions, under which at least one of the ammonium salts is partly melted.

2. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, allowing the mixture to cool down and solidify, adding to the solid mixture of ammonium salts another fertilizing agent and heating to a temperature at which at least one of the ammonium salts is partly melted.

3. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, allowing the mixture of ammonium salts to cool down and solidify, adding to it another fertilizing agent heated to a temperature, at which one of the ammonium salts is partly melted, applying heat to effect a partial melting of the mixture and allowing same to cool down and solidify.

4. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric

acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, allowing the mixture of ammonium salts to cool down and solidify, adding to it another fertilizing agent under conditions under which one of the ammonium salts is partly melted, and passing ammonia gas in contact with the mixture while the same is allowed to cool down and solidify.

5. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, allowing the mixture to solidify, adding to the solid mixture of ammonium salts a potassium salt having fertilizing properties and heating to a temperature at which at least one of the ammonium salts is partly melted.

6. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, supplying heat and removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, allowing said mixture to cool down and solidify, adding to the solid mixture of ammonium salts another fertilizing agent and heating to a temperature at which at least one of the ammonium salts is partly melted.

7. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, adding to this mixture of ammonium salts a calcium salt having fertilizing properties and heating to a temperature at which at least one of the ammonium salts is partly melted.

8. The method of producing a fertilizer comprising simultaneously introducing into a hot molten body of ammonium nitrate and ammonium phosphate dilute nitric acid, phosphoric acid and ammonia gas in such proportion as to effect substantial neutralization of the acids, removing the mixture of ammonium nitrate and ammonium phosphate from the reaction zone, adding to this mixture of ammonium salts a clayey substance having fertilizing properties and heating to a temperature at which at least one of the ammonium salts is partly melted.

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