

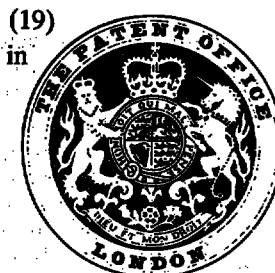
PATENT SPECIFICATION

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(54) GRANULAR AMMONIUM SULPHATE

(71) We, AUSTRALIAN FERTILIZERS LIMITED, a company incorporated under the laws of the State of Victoria, Commonwealth of Australia, of 213 Miller Street, North Sydney, in the State of New South Wales, Australia, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention relates to the production of granular ammonium sulphate.

Ammonium sulphate is commonly crystallized from solutions produced as a by-product from coke ovens and caprolactam plants. It may also be prepared in crystalline form by the direct ammoniation of sulphuric acid in reactor crystallisers, or by reacting gypsum and ammonium carbonate as in the Merseberg process.

However, crystalline ammonium sulphate is generally fine, poorly sized and not free flowing. These physical properties create problems in the bulk blending of fertilizers containing ammonium sulphate, and in the spreading of ammonium sulphate and fertilizers containing this product in a blended form.

Attempts have been made to overcome this problem by introducing new techniques in the re-crystallization processes, an example of this being the method of counter-current crystallization.

Formerly, the crystalline particles growing by evaporation in vacuum crystallisers were led in numerous steps from the hot to the next cooler stage. In the newer counter-current processes, the fine crystals formed during a cooler stage are led to the next warmer stage where they are mixed with an evaporating hot solution. Here the colder crystals serve as nuclei for the formation of crystals from the over-saturated solution due to evaporation of the next (warmer)

stage.

The aforesaid counter-current process can be controlled to yield a substantial fraction of coarse crystals that can be recovered by screening. These crystals are rounded and are nearly spherical in shape and are more favoured by consumers for use in blending of mixed fertilizers. However, the fuel usage and maintenance costs in the dissolution and recrystallization processes are relatively high and lead to higher production costs for this product.

Also, recovery of ammonium sulphate fines left over from processing operations such as screening may lead to problems. For example, fine material may be agglomerated by compaction. Compaction is carried out by passing the said fine material between rolls. In one method of compaction two smooth rolls are used, which give a continuous cake which is then broken up and screened to give the required size range. The fines and the crushed oversize material are fed back to the compaction plant. Another method of compaction utilises one grooved roll and one smooth roll. This has the advantage of less crushing of the material. The product from the rolls is passed over the screens which eliminate the fines which have not been compacted. The oversize particles and the fines are fed back to the compaction process.

The compacted product from these processes may be then conveyed to a rotating drum where the edges of the particles are smoothed off by attrition.

Generally, the products made by the above fines recovery processes are formed of particles which are angular and irregular in shape, and many consumers prefer a more rounded particle, for use in the manufacture of mixed fertilizers.

Attempts have been made to overcome this problem by granulating ammonium sulphate e.g. the reaction product of ammonia

and sulphuric acid. A common method of granulation is to use a rotary ammoniator granulator, as developed by the Tennessee Valley Authority. Another common method is to use a pugmill to contact recycle fines with varying proportions of ammonia and sulphuric acid using as granulation aids, phosphates, ammonium nitrate, urea or lignosulphates. It has been found that these methods of granulating ammonium sulphate tend to produce a product which is not hard and free flowing at economic levels of usage of granulating aids.

In accordance with the present invention there is provided a process for the production of granular ammonium sulphate not containing ammonium nitrate characterised in that ammonium sulphate is granulated in the presence of a granulation aid which is an aluminium salt or a ferric salt.

Preferably, in the process of the present invention, ammonium sulphate is distributed onto a bed of ammonium sulphate fines and is then granulated in the presence of the aluminium salt or the ferric salt. The ammonium sulphate may be contained in a slurry formed by partially or wholly ammoniating sulphuric acid, and in this case the slurry is distributed onto the bed of fines. Preferably, the fines are recycled fines.

The granulation may be carried out in known types of apparatus such as rotary granulators or pugmills.

Preferably, the aluminium salt is aluminium sulphate. Ferric ammonium sulphate is preferred as the ferric salt. However it has been found that ferric salts are less effective than aluminium salts at similar levels of concentration. It has been found that a convenient way of introducing the aluminium or ferric salt to the ammonium sulphate is in the form of an aqueous solution. This may be metered into the granulation apparatus at a convenient location such as adjacent the inlet thereof.

Alternatively the required amount of aluminium or ferric salt, in the solid form, may be added to the recycle or granulator, by means of a feeding apparatus.

Where an aluminium salt is used, it is preferably used in an amount such as to give a final product containing from 0.4 to 2.0% by weight of the aluminium salt measured as Al_2O_3 . It is preferred to not use a concentration higher than the upper end of this range so as to avoid unduly reducing the nitrogen content of the final product. Further, below the preferred range quoted above the improvement in granulation obtained is less marked. More preferably the concentration of aluminium salt in the final product is 0.5 to 1.0% by weight of aluminium salt measured as Al_2O_3 .

The granulation product produced by the process of the present invention may be

allowed to self-dry under ambient conditions or it may be dried in conventional equipment.

The product run may contain phosphate derived from the starting materials or phosphate may be deliberately added. The presence of phosphate does not significantly affect the granulation of the ammonium sulphate. Further, a potassium salt may also be added to the product run.

It has been found that granulated ammonium sulphate which has been granulated in the presence of an aluminium or ferric salt as described hereinabove (and products, such as fertilizers, containing such granulated ammonium sulphates) are free flowing and do not consolidate or set to a hard mass on standing. Further, one embodiment of the present invention described above enables ammonium sulphate fines to be recovered by recycling them through the granulation apparatus and forming them into a bed onto which a slurry containing ammonium sulphate is distributed.

In accordance with a preferred form of the present invention, a process for the production of granular ammonium sulphate is characterised in that sulphuric acid is partially neutralised with ammonia to form a slurry containing ammonium sulphate such that a 10% solution of the slurry has a pH from 1.5 to 1.7, the slurry is distributed onto a bed of ammonium sulphate fines and is then subjected to further ammoniation simultaneously with the granulation of the ammonium sulphate, in the presence of a granulation aid which is an aluminium or ferric salt, so as to produce a product containing granulated ammonium sulphate and having a pH of from 4.0 to 4.5 as a 10% solution.

The present invention will now be illustrated by the following Examples:

Example 1.

Ammonia and sulphuric acid were reacted in a vessel so that the pH of a 10% solution of the resultant slurry was 1.5 to 1.7.

The slurry was distributed onto a bed of recycled ammonium sulphate fines in a rotary granulator and sufficient commercial grade aluminium sulphate aqueous solution (7.5% by weight Al_2O_3) was metered into the inlet end of the granulator to give a final content in the product of 0.75% by weight aluminium sulphate measured as Al_2O_3 . The slurry was granulated in the granulator while simultaneously further ammoniation was carried out to give a final product pH (of a 10% solution) of 4.5.

The product was then dried to a moisture content of 0.5% by weight and screened to remove over and under sized material. The oversized material was crushed and returned with the fines to the recycle.

For a production rate of 5 tons per hour, a recycle rate of 30 tons per hour was maintained. The final product had a typical analysis of 20.5%N.

The product when stored in bulk or bags did not consolidate or set to a hard mass even though the material was not after treated to prevent this commonly occurring property.

Example 3.

200 g of crystalline ammonium sulphate was sprayed with ferric ammonium sulphate solution in a 12" diameter drum of a pilot plant scale rotary granulator. The drum was rotated at 40% of the critical speed for 4 minutes.

The resulting granules were dried at 90°C to 0.5% moisture, yielding a hard, free flowing granular product. The ferric ammonium sulphate content of the final product was 1.5% by weight measured as ferric oxide and the nitrogen (N) content was 20.0% by weight.

WHAT WE CLAIM IS:-

1. A process for the production of granular ammonium sulphate not containing ammonium nitrate characterised in that ammonium sulphate is granulated in the presence of a granulation aid which is an aluminium or a ferric salt.

2. A process as claimed in Claim 1 characterized in that the ammonium sulphate is distributed onto a bed of ammonium sulphate fines and is then granulated.

3. A process as claimed in Claim 2 characterized in that the fines are recycled fines.

4. A process as claimed in Claim 2 or Claim 3 characterized in that the ammonium sulphate is contained in a slurry of partially or wholly ammoniated sulphuric acid which is distributed onto the bed of ammonium sulphate fines and is then subjected to further ammoniation simultaneously with the granulation of the ammonium sulphate.

5. A process as claimed in any one of Claims 1 to 4 characterized in that the

aluminium salt is aluminium sulphate.

6. A process as claimed in Claim 5 characterized in that the aluminium salt is used in an amount such as to give a final product containing from 0.4 to 2.0% by weight of the aluminium salt measured as Al_2O_3 .

7. A process as claimed in Claim 6 characterized in that the aluminium salt is used in an amount such as to give a final product containing from 0.5 to 1.0% by weight of the aluminium salt measured as Al_2O_3 .

8. A process as claimed in any one of the preceding claims characterized in that phosphate is also present during the granulation of the ammonium sulphate.

9. A process as claimed in any one of the preceding claims characterized in that a potassium salt is also present during the granulation of the ammonium sulphate.

10. A process as claimed in Claim 1 for the production of granular free flowing ammonium sulphate substantially as herein described.

11. A process for the production of granular ammonium sulphate characterized in that sulphuric acid is partially neutralised with ammonia to form a slurry containing ammonium sulphate such that a 10% solution of the slurry has a pH from 1.5 to 1.7, the slurry is distributed onto a bed of ammonium sulphate fines and is then subjected to further ammoniation simultaneously with the granulation of the ammonium sulphate in the presence of a granulation aid which is an aluminium salt or a ferric salt so as to produce a product containing granulated ammonium sulphate and having a pH of from 4.0 to 4.5 as a 10% solution.

12. Free flowing granular ammonium sulphate whenever produced by a method as claimed in any one of the preceding claims.

13. A fertiliser containing ammonium sulphate as claimed in Claim 12.

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Agents for the Applicants.



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3. A process as claimed in Claim 2 characterized in that the fines are recycled fines.

4. A process as claimed in Claim 2 or Claim 3 characterized in that the ammonium sulphate is contained in a slurry of partially or wholly ammoniated sulphuric acid which is distributed onto the bed of ammonium sulphate fines and is then subjected to further ammoniation simultaneously with the granulation of the ammonium sulphate.

5. A process as claimed in any one of Claims 1 to 4 characterized in that the

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8. A process as claimed in any one of the preceding claims characterized in that phosphate is also present during the granulation of the ammonium sulphate.

9. A process as claimed in any one of the preceding claims characterized in that a potassium salt is also present during the granulation of the ammonium sulphate.

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