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**(12) PATENT ABRIDGMENT      (11) Document No. AU-B-30059/89**  
**(19) AUSTRALIAN PATENT OFFICE      (10) Acceptance No. 618579**

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(54) Title  
**PROCESS FOR THE SUPPRESSION OF DUST AND FINES DURING THE GRANULATION OF AMMONIUM NITRATE WITH A NITROGEN CONTENT OF 28% - 34.5%**

International Patent Classification(s)  
(51)<sup>4</sup> **C05C 001/02**

(21) Application No. : **30059/89**

(22) Application Date : **17.02.89**

(30) Priority Data

(31) Number	(32) Date	(33) Country
<b>3811913</b>	<b>09.04.88</b>	<b>DE FEDERAL REPUBLIC OF GERMANY</b>

(43) Publication Date : **12.10.89**

(44) Publication Date of Accepted Application : **02.01.92**

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(56) Prior Art Documents  
**EP 207222**  
**GB 373211**  
**US 3867124**

(57) Claim

1. A process for suppressing fines, during the granulation of ammonium nitrate having a nitrogen content of 28% to 34.5%, comprising the steps of:

A) granulating ammonium nitrate, to produce a raw granulate containing fines; and

B) simultaneously performing the steps of:

i) heating the raw granulate and fines to a temperature from 100°C to 125°C;

ii) adding to the raw granulate and fines a hydrated material, selected from the group consisting of divalent iron sulfate, trivalent iron sulfate, and magnesium sulfate, in an amount sufficient such that the sulfate concentration is from 0.1% to 1.0% by weight; and

iii) granulating the raw granulate and fines, wherein a substantial amount of the fines produced in step A are affixed to the raw granulate.

AUSTRALIA

618579

PATENTS ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE

Application Number:  
Lodged:

Complete Specification Lodged:  
Accepted:  
Published:

Priority:  
Related Art:

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TO BE COMPLETED BY APPLICANT

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Complete Specification for the invention entitled Process for  
the suppression of dust and fines during the granulation of  
ammonium nitrate with a nitrogen content of 28% - 34.5%.

The following statement is a full description of this invention  
including the best method of performing it known to me:-

The invention relates to a process for the suppression of dust and fines during the granulation of ammonium nitrate with a nitrogen content of 28% - 34.5%.

Ammonium nitrate for use in fertilizer production should consist as far as possible of spherical granules with a grain size ranging between approx. 1 - 4 mm diameter. As every granulation process also produces granules outside the range of 1 - 4 mm, a special screening stage is necessary to eliminate fines and oversize, the latter being crushed and fed back to the granulator together with the fines. Granulation processes are thus cyclical in nature for a proportion of the substance and so dependent in terms of both equipment and power requirement on the quantity of substance in the return cycle.

It is true that dual-stage granulation processes are already known, yet these are normally the result of combining two essentially different granulation systems, e.g. prilling and drum granulation, or drum granulation and fluidized-bed granulation. Patents DE-2164731 and FR-7246363, for example, describe the grain enlargement of AN fertilizers by diverting a part-stream of the melt upstream of the prilling stage, which is then sprayed onto the prills in a drum granulator. The prills are required as a starting product before being enlarged to form the end product by the addition of further melt.

Even so, the extent to which the formation of outsize can be suppressed by the provision of optimum granulating conditions (e.g. liquid phase, granulation temperature and use of additives) is limited.

The purpose of the invention is to provide a solution in

the form of a granulation loop for ammonium nitrate with a very narrow grain spectrum, i.e. to produce as large a quantity of ammonium nitrate as possible within a diameter range of 1 - 4 mm. The outsize, especially the fines, are to be suppressed.

10 In the case of a process such as that described above, this task is fulfilled in terms of the invention in that the raw granulate is formed in a first granulation zone based on a known granulation process using screw, drum, spherodizer and/or plate granulators; whilst the dust and/or fines formed in the first granulation zone are fixed to the raw granulate in a second granulation zone at a temperature of between 100°C and 125°C by the addition of di- or trivalent iron sulphate containing an appropriate quantity of water of crystallization.

It has been proven that the production of ammonium nitrate granules within a narrow grain spectrum is possible by addition of iron sulphate containing water of crystallization, the proportion of iron sulphate ( $\text{FeSO}_4$ ) being 0.1% - 1.10%/wt of the quantity to be produced. As a result of the melting that takes place in the water of crystallization present, a second liquid phase is caused which permits the granulation of the remaining dust and fines.

As a result of the process specified in the invention, the proportion of fines in the second granulation zone is significantly reduced. The quantity of fines in the return cycle is thus reduced proportionately.

The reduced quantity of fines in the return cycle has the corollary of reduced equipment expenditure and power requirement.



The configuration specified in the invention provides that an equivalent additive, e.g.  $\text{MgSO}_4 \times \text{H}_2\text{O}$  be admixed. This salt melts slowly in the water of crystallization at the granulation temperature.

Especially effective results are obtained if both granulation zones are combined in a single granulator.

However, even better results are obtained than in conventional processes to date if, in further embodiments of the invention, a plate granulator is followed by a drum granulator. Furthermore, it is possible within the terms of the invention to combine the second granulation zone with a drying zone.

The invention is further elucidated below by means of examples, which in no way restrict the above claims.

#### Example 1

A production unit yields alternatively calcium ammonium nitrate with 26% - 27.5% N; and ammonium nitrate with 33.5% N. The starting product for the ammonium nitrate production (without admixture of iron sulphate in the granulator) is characterized as follows:

N content:	33.5%
H <sub>2</sub> O content:	0.2% (Karl-Fischer method)
Granulation additive:	0.83%
Grain hardness:	3.6 kg

The results of a screen analysis (in % wt) are shown below:

	Product	After Granulation	Return Cycle
+ 5 mm	0	11.4	0
+ 4 mm	0.5	15.1	1.0
+ 3.15 mm	24.4	21.6	4.2
+ 2 mm	96.0	56.2	26.3
+ 1 mm	99.0	88.3	76.7
+ 0.5 mm	100.0	97.3	96.5
- 0.5 mm	0	2.7	3.5
Total	100.0	100.0	100.0

The onsize (2 - 4 mm) constitutes 41.1% (56.2 - 15.1) and the fines ( 1 mm) 11.7% (100 - 88.3) after granulation.

#### Example 2

Using the same ammonium nitrate production process in the same unit, but with the addition of iron sulphate in the granulator, the starting product is characterized as follows:

N content:	33.5%
H <sub>2</sub> O content:	0.25% (Karl-Fischer method)
Granulation additive:	0.98% (including FeSO <sub>4</sub> )
Grain hardness:	3.3 kg

The results of a screen analysis (in % wt) are shown below:

	Product	After Granulation	Return Cycle
+ 5 mm	0	7.4	0
+ 4 mm	6.2	15.7	1.5
+ 3.15 mm	45.2	36.5	9.9
+ 2 mm	99.2	81.5	47.7
+ 1 mm	100.0	98.8	87.6
+ 0.5 mm	0	99.5	98.3
- 0.5 mm	0	0.5	1.7
Total	100.0	100.0	100.0

The onsize constitutes 65.8% (81.5 - 15.7) and the fines 1.2% (100 - 98.8) after granulation.

The proportion of fines has been reduced by almost 90% as against example 1 with a proportionate increase in the required grain size. On account of the increased proportion of required grain size, a greater proportion will be removed from the cycle during screening, thereby reducing the quantity in the return cycle. In order to maintain the optimum temperature and granulation conditions in the liquid phase, a certain quantity of the product is added to the return cycle prior to cooling. The overall quantity in the return cycle is still less than when no iron sulphage is admixed. The capacity of the production unit can thus be increased accordingly and power saved at the drying stage.

The claims defining the invention are as follows:

1. A process for suppressing fines, during the granulation of ammonium nitrate having a nitrogen content of 28% to 34.5%, comprising the steps of:

A) granulating ammonium nitrate, to produce a raw granulate containing fines; and

B) simultaneously performing the steps of:

i) heating the raw granulate and fines to a temperature from 100°C to 125°C;

ii) adding to the raw granulate and fines a hydrated material, selected from the group consisting of divalent iron sulfate, trivalent iron sulfate, and magnesium sulfate, in an amount sufficient such that the sulfate concentration is from 0.1% to 1.0% by weight; and

iii) granulating the raw granulate and fines, wherein a substantial amount of the fines produced in step A are affixed to the raw granulate.

2. The process for suppressing fines, according to Claim 1, wherein the hydrated material of step Bii is divalent, iron sulfate or trivalent iron sulfate.

3. The process for suppressing fines, according to Claim 1, wherein the hydrated material of step Bii is magnesium sulfate.

4. The process for suppressing fines, according to Claim 2, wherein steps A and B are conducted in a singular granulating device.

5. The process for suppressing fines, according to Claim 3, wherein steps A and B are conducted in a singular granulating device.

6. The process for suppressing fines, according to Claim 2, wherein step A is conducted utilizing a plate granulator and step B is conducted utilizing a drum granulator.

7. The process for suppressing fines, according to Claim 3, wherein step A is conducted utilizing a plate granulator and step B is conducted utilizing a drum granulator.



8. The process for suppressing fines, according to Claim 2, wherein step B includes the simultaneous step of drying.

9. The process for suppressing fines, according to Claim 3, wherein step B includes the simultaneous step of drying.

DATED this 15th day of October, 1991.

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By Its Patent Attorneys  
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