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(54) **A Process for the Production of
Urea Granules**

(57) The production of urea granules
by prilling or granulating a urea melt
or an aqueous urea solution, which
melt or solution contains a

magnesium oxide containing additive.
The urea granules thus obtained
exhibit a high crushing strength, a
high apparent density, and a very low
caking tendency, and are suitable for
bulk blending with single and triple
superphosphate granules.

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SPECIFICATION

A Process for the Production of Urea Granules

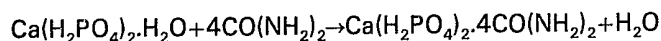
This invention relates to a process for the production of urea granules.

For the production of urea granules, various methods are known. One of these is prilling, by which
 5 in this specification is understood a method in which a substantially anhydrous urea melt (having a
 water content of no more than 0.1 to 0.3 % by weight) is sprayed in the top of a prilling column in a
 rising stream of air of ambient temperature, in which the droplets solidify. The resulting prills have a
 maximum diameter of not much more than 3 mm, and are mechanically rather weak.

Urea granules having larger dimensions and better mechanical properties can be produced by the
 10 granulation of a substantially anhydrous urea melt in a drum generator, for example, by the sledrozier
 technique as described in British Patent Specification No. 894,773, or in a pan granulator, for example
 as described in U.S. Patent Specification No. 4,008,064, or by the granulation of an aqueous urea
 solution in a fluidized bed, for example as described in Dutch patent application 7806213. In the
 process described in the last-mentioned publication, an aqueous urea solution having a urea
 15 concentration of 70—99.9% by weight, preferably 85—96% by weight, is sprayed in the form of very
 fine droplets having an average diameter of 20—120 micron into a fluidized bed of urea particles at a
 temperature at which the water evaporates from the solution sprayed onto the particles, and urea
 solidifies on the particles to form granules having a desired size which may be 25 mm and more. As
 rather large amounts of flydust are formed in this process, especially if the urea solution used as the
 20 starting material contains more than 5% by weight of water, in particular more than 10% by weight of
 water, preferably a crystallization retarder for the urea, in particular a water-soluble addition or
 condensation product of formaldehyde and urea, is added to the urea solution, whereby the formation
 of flydust is suppressed practically completely. The result of the presence of the crystallization retarder
 is that the granules remain plastic as they are being formed, so that owing to rolling and/or impacts
 25 during the formation mechanically strong, smooth and round granules may be formed.

The resulting granules have a high crushing strength, a high impact resistance, and little tendency
 of forming flydust through abrasion, and moreover do not cake together, not even upon prolonged
 storage, although urea exhibits a strong natural tendency of caking together.

Urea granules produced according to one of the known methods cannot be used for the
 30 production of heterogeneous binary and ternary fertilizer mixtures, such as N-P or N-P-K mixtures by
 bulk blending with the cheap superphosphate or triple superphosphate, as such urea granules are
 incompatible with these phosphates. Mixtures of such urea granules with superphosphate or triple
 superphosphate granules deliquesce after some time to form an unmanageable and unusable mud.
 According to a paper by G. Hoffmeister and G. H. Megar, presented during "The Fertilizer Industry
 35 Round Table" on November 6, 1975, at Washington D.C., this incompatibility is caused by a reaction
 according to the following equation



By the reaction of 1 mol monocalcium phosphate monohydrate, the main component of
 superphosphate and triple superphosphate, with 4 mols urea, a urea-monocalcium phosphate adduct
 40 is formed, with 1 mol water being released. As the adduct has a high solubility, it is readily dissolved in
 the water liberated to form a large volume of solution, which wets the granules in the mixture, owing to
 which the reaction proceeds at an ever faster rate. Up until now, no commercially acceptable means
 have been found to make urea compatible with superphosphate or triple superphosphate. As a
 consequence, it is virtually only the more expensive phosphate fertilizers monoammonium phosphate
 45 and diammonium phosphate that can be used for bulk blending with urea.

It is an object of the present invention to provide a process for the production of urea granules
 having preferred properties, including compatibility with superphosphate and triple superphosphate
 granules.

The invention provides a process for the production of urea granules by prilling or granulating a
 50 urea melt or an aqueous urea solution, which process is characterized in that the urea melt or solution
 to be prilled or granulated contains a magnesium oxide containing additive.

It has surprisingly been found that the granules produced according to the invention are
 compatible with superphosphate and triple superphosphate granules, by virtue of which they are
 suitable for bulk blending with these phosphate fertilizers. Mixtures of urea granules obtained
 55 according to the invention with superphosphate granules or triple superphosphate granules that were
 subjected to the "TVA Bottle Test" at 27°C were still dry after 7 weeks, whereas analogous mixtures
 with urea granules not produced in accordance with the invention had deliquesced completely after 3
 days.

It has further been found that the presence of magnesium oxide during the prilling or granulation
 60 of a urea melt or solution has for its result that the building-up of the granules proceeds flawlessly, and
 the formation of flydust is prevented, while in addition the resulting urea granules have a very high
 crushing strength and a very high apparent specific gravity. A further highly surprising feature is that

the urea granules produced according to the invention do not cake together, not even upon prolonged storage.

The magnesium oxide can be used as such (MgO) or in the form of fully calcined dolomite (MgO+CaO) or a selectively calcined dolomite (MgO+CaCO₃). A beneficial effect is already observed with a quantity of additive corresponding to 0.1% by weight of MgO, calculated on the urea in the melt or solution. Preferably the additive is used in a quantity corresponding to 0.5—2% by weight of MgO, calculated on the urea in the melt or solution. If desired, higher proportions may be used, but this does not seem to offer any particular advantages. The additive may be added in the form of a powder to the urea melt or solution prior to prilling or granulating.

Preferably, after their formation the granules are cooled to 30°C or to a lower temperature, for example by means of a stream of air, the moisture content of which has preferably been reduced to such an extent that during the cooling process the granules do not absorb moisture from the cool air.

The invention also relates to compatible, heterogeneous fertilizer mixtures of urea granules obtained by the process according to the present invention with superphosphate or triple superphosphate granules, and, if desired, one or more other granular substances.

In addition to urea and superphosphate or triple superphosphate, a potassium fertilizer is mostly included in the mixture, such as KCl. To prevent segregation of the mixture, the granule dimensions of the components to be blended must be adapted to one another.

For further information regarding the production of fertilizer granules, reference is made with regard to prilling to U.S. Patent Specification No. 3,130,225 with regard to granulation in a pan generator to U.S. Patent Specification No. 4,008,064, with regard to granulation in a drum generator to British Patent Specification No. 894,773 and with regard to granulation in a fluidized bed to Dutch patent application 7806213.

The effect of the process according to the invention is shown in and by the following examples. The "TVA Bottle Test" referred to in the examples serves to determine the compatibility of urea granules with superphosphate and triple superphosphate granules. In this test, a mixture of the urea granules to be tested with superphosphate or triple superphosphate granules was kept in a closed bottle of 120 cm³ at 27°C, and the condition was periodically inspected. The condition observed is evaluated as follows:

30 Condition of the mixture

- D=dry, free-flowing
 W-1=moisture patches, but usable
 W-2=moist and slightly sticky, but probably usable
 W-3=wet through and sticky, unsuitable for use
 W-4=very wet, unsuitable for use
 H=hard caked together, unsuitable for use.

In the "bag test" referred to in the examples, the caking tendency of the granules tested was determined. In this test urea granules were packed in bags of 35 kg, which were stored under a weight of 1000 kg at 27°C. After 1 month, the average number of lumps per bag was determined, and the average hardness of the lumps was measured. By hardness is understood here the force in kg, exercised by a dynamometer to cause a lump of 7×7×5 cm to disintegrate.

The crystallization retarder F 80, referred to in the examples, is a clear viscous liquid commercially available under the name of "Formurea 80", which is stable between -20°C and +40°C, and upon analysis is found to contain per 100 parts by weight approximately 20 parts by weight of water, approximately 23 parts by weight of urea and approximately 57 parts by weight of formaldehyde, approximately 55% of the formaldehyde being bound as trimethyl urea, and the balance being present in the unbound state.

Example I

Tests were conducted in which an aqueous urea solution with and without a known crystallization retarder (F 80) and with magnesium oxide as a crystallization retarder was sprayed into a fluidized bed of urea particles. The granulation conditions and the physical properties of the resulting granules are listed in Table A.

Table A

Crystallization retarder	none	1% F80	0.6 MgO	1% MgO
<i>Granulation conditions</i>				
urea solution				
concentration, % by weight	94.6	94.5	94.5	94.5
temperature, °C	130	130	130	130
rate of flow, kg/h	280	280	280	280

Table A (cont.)

		Crystallization retarder	none	1%F80	0.6 MgO	1% MgO	
5	injection air						5
	rate of flow, Nm ³ /h		130	130	130	130	
	temperature, °C		140	140	140	140	
	fluidization air						
	rate of flow, Nm ³ /h		850	850	850	850	
	temperature, °C		45	64	54	58	
	bed temperature, °C		108	105	104	104	
10	<i>Product properties</i>						10
	apparent density, g/cm ³		1.23	1.26	1.30	1.31	
	crushing strength dia. 2.5 mm, kg		2.1	2.8	4.0	4.3	
	fly dust, g/kg		5.4	<0.1	0.1	<0.1	
15	bag test						15
	lumps, %		100	10	0	0	
	hardness, kg		22	0.1	0	0	
TVA Bottle Test with superphosphate (SSP) 50/50							
20	after 1 day		W-2	W-2	D	D	20
	after 3 days		W-4	W-4	D	D	
	after 7 days		W-4	W-4	D	D	
	after 14 days		W-4	W-4	W-1	D	
	after 21 days		W-4	W-4	W-1	D	
	after 50 days		W-4	W-4	W-1	D	
with triple superphosphate (TSP) 50/50							
25	after 1 day		W-2	W-2	D	D	25
	after 3 days		W-4	W-4	D	D	
	after 7 days		W-4	W-4	D	D	
	after 14 days		W-4	W-4	W-1	D	
	after 21 days		W-4	W-4	W-1	D	
30	after 50 days		W-4	W-4	W-1	D	30

Example II

A subsequent series of tests were conducted analogously to those described in Example I, but using selectively calcined dolomite and completely calcined dolomite as crystallization retarders instead of magnesium oxide. The granulation conditions and the physical properties of the resulting granules are listed in Table B.

Table B

		Crystallization retarder	none	<i>selectively calcined dolomite</i>		<i>completely calcined dolomite</i>		
				1.5%	3%	1.5%	3%	
40	<i>Granulation conditions</i>							40
	urea solution							
	concentration, % by weight		94.5	95.5	95.5	95.5	95.5	
	temperature, °C		130	130	130	130	130	
	rate of flow, kg/h		200	220	220	220	220	
45	injection air							45
	rate of flow, Nm ³ /h		130	130	130	130	130	
	temperature, °C		140	134	149	150	149	
50	fluidization air							50
	rate of flow, Nm ³ /h		850	850	850	850	850	
	temperature, °C		58	75	66	53	57	
	bed temperature, °C		100	98	92	95	94	

Table B (cont.)

		<i>completely calcined dolomite</i>		<i>selectively calcined dolomite</i>			
5	Crystallization retarder	none	1.5%	3%	1.5%	3%	
	<i>Product properties</i>						
	apparent density, g/cm ³	1.22	1.29	1.31	1.29	1.32	5
	crushing strength, dia.2.5mm/kg	1.9	4.0	3.9	3.7	3.4	
	fly-dust, g/kg	2.2	0	0	0	0	
10	bag test						
	lumps, %	100	9	7	10	23	10
	hardness, kg	13	<1	<1	2	2	
	TVA Bottle Test						
	with SSP 50/50	after 3 days deliquesced	14 days usable	14 days usable	more than 60 days usable	more than 60 days usable	
15	with TSP 50/50	after 3 days deliquesced	14 days usable	14 days usable	more than 60 days usable	more than 60 days usable	15

Example III

20 A substantially anhydrous urea melt with and without added magnesium oxide was sprayed in the top of a prilling column in a rising stream of air of ambient temperature. The physical properties of the resulting prills are listed in Table C. 20

Table C

		none	0.72% MgO	0.95% MgO	
25	<i>Product properties</i>				
	apparent density, g/cm ³	1.30	1.32	1.33	25
	crushing strength, dia. 2.5 mm, kg	0.54	1.04	1.16	
	bag test				
	lumps, %	100	0	0	
	hardness, kg.	9	0	0	
30	TVA Bottle Test				30
	with SSP 50/50	after 3 days deliquesced	more than 60 days usable	more than 60 days usable	
	with TSP 50/50	after 3 days deliquesced	more than 60 days usable	more than 60 days usable	

35 Example IV

40 A urea melt to which magnesium oxide had been added was granulated in a rotary horizontal granulation drum having a diameter of 90 cm and a width of 60 cm. The drum was provided on its inner wall with eight longitudinal strips of 3.5×60 cm spaced equal distances from each other. The speed was 15 rpm. The drum was filled with 60 kg urea granules having an average diameter of 1.8 mm and a temperature of 80°C. Using two hydraulic sprayers, 60 kg anhydrous urea melt (99.8% by weight of urea), to which 0.6% by weight of MgO had been added, having a temperature of 140—145°C was sprayed into the rotary drum at a rate of approximately 100 kg/h over the granules showered from the longitudinal strips. The granulation was effected at 110°C. 40

45 At the end of the test, the granules were cooled to approximately 30°C and sieved. The product granules had a good roundness and a smooth surface. Their apparent density was 1.288 g/cm³ and the crushing strength dia. 2.5 mm was 3.5 kg. Flydust formation was 3.9 g/kg. The granules had virtually no caking tendency. 50/50 blends with SSP and with TSP were usable for more than 60 days. The sieve analysis of the product was as follows: 45

50	>4.00 mm:	17%			
	4.00—2.5 mm:	46%			50
	2.5—2.0 mm:	29%			
	<2.0 mm:	8%			
	average diameter:	3.0 mm.			

Claims

1. A process for the production of urea granules or urea coated granules, wherein the granule forming or growing, urea containing liquid incorporates or contains magnesium oxide.
2. A process for the production of urea granules by prilling or granulating a urea melt or an aqueous urea solution, characterized in that the urea melt or solution to be prilled or granulated contains a magnesium oxide containing additive. 5
3. A process as claimed in claim 2, wherein the additive is magnesium oxide *per se*.
4. A process as claimed in claim 1 or 2, wherein the magnesium oxide is incorporated or contained in the form of selectively or completely calcined dolomite.
5. A process as claimed in any one of claims 1 to 4, wherein the amount of additive corresponds to at least 0.1% by weight of MgO calculated on the urea in the melt or solution. 10
6. A process as claimed in claim 5, wherein the amount of additive corresponds to from 0.5 to 2% by weight of MgO, calculated on the urea in the melt or the solution.
7. A process for the production of urea granules as defined in claim 2, and substantially as hereinbefore described with reference to any one of the foregoing individual examples I to IV. 15
8. Urea granules produced by a process as claimed in any one of the preceding claims.
9. A compatible heterogeneous fertilizer blend comprising urea granules produced by a process as defined in any one of claims 1 to 7 and single or triple superphosphate granules.
10. A blend as claimed in claim 9, further comprising a granular fertilizer such as KCL.
11. Urea granules containing or incorporating magnesium oxide. 20
12. Granules having a coating of urea which contains or incorporates magnesium oxide.
13. The features hereinbefore disclosed or their novel equivalents in any combination.

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