

[54] **PRODUCTION OF GRANULAR SODIUM
DICHLOROISOCYANURATE**

3,548,046 12/1970 Savage 264/140

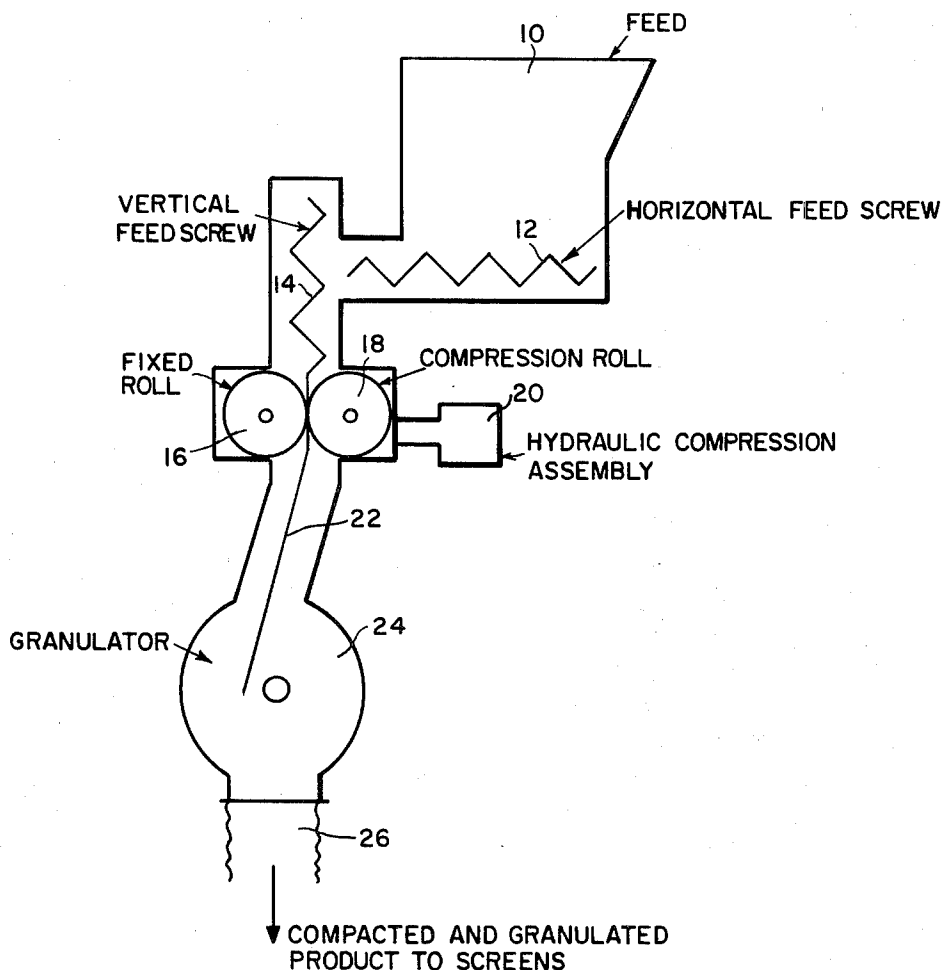
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Assistant Examiner—T. E. Balhoff[73] Assignee: **FMC Corporation**, New York, N.Y.[57] **ABSTRACT**[22] Filed: **Mar. 19, 1973**[21] Appl. No.: **342,511**[52] U.S. Cl. **264/118; 264/140; 264/145**[51] Int. Cl. **B28b 11/14**

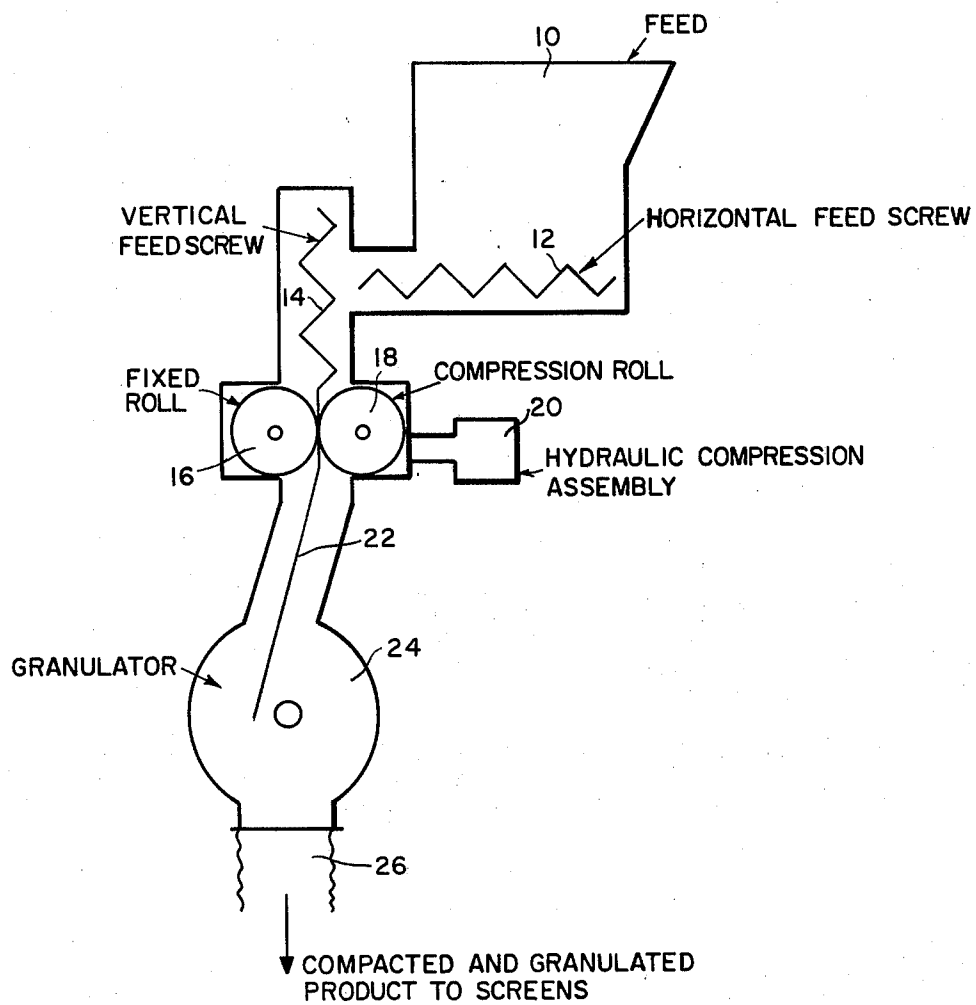
[58] Field of Search 264/118, 140, 144, 145

Granular sodium dichloroisocyanurate is made by forming sheets of sodium dichloroisocyanurate containing from 11.0 to 14% of combined water, by feeding sodium dichloroisocyanurate powder between sets of rolls at pressures of the order of $7,500 \pm 50\%$ pounds of force per linear inch of contact line, breaking up the sheets to produce a mixture of powder and granules, and then screening the broken-up mixture to get the desired granules. The granular material may be dried, if desired, to anhydrous granules, or used in hydrated form.

[56] **References Cited****UNITED STATES PATENTS**

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1 Claim, 1 Drawing Figure



PRODUCTION OF GRANULAR SODIUM DICHLOROISOCYANURATE

This invention relates to the production of granular sodium dichloroisocyanurate from the powdery solids obtained in the manufacturing process.

Sodium dichloroisocyanurate is widely used as a source of available chlorine in solid bleach and detergent compositions, and in chlorination of water in swimming pools. It is desirably sold as granules (20 to 70 mesh — U.S. standard screen). Since it is generally recovered from manufacturing operations as a powder in the 200 to 400 mesh size range, it is necessary to pelletize the fine material. This may be done by high-pressure tableting, followed by crushing, or by sheeting in a roll compactor into corrugated, readily frangible sheets, which are then broken down to granules in any convenient machine.

Both processes have disadvantages. In tableting, anhydrous sodium dichloroisocyanurate sticks to the molds, so that it was considered necessary to add a mold lubricant. However, in accordance with Japanese patent 1967-23198, published Nov. 10, 1967, the adhesion of the powder to the mold can be overcome by first adding 4 to 14% of water to the anhydrous material. The resultant hydrate does not stick to the mold, even at the required pressure of 1,000 kilograms per square centimeter (equivalent to about 14,200 pounds per square inch).

In the sheeting or compaction process, other problems arise. A principal problem is that of excessive fines production, requiring recycle rates of 60 to 80%, where a +70 mesh granule is desired. A second problem is that temperature control is essential in the process, since anhydrous sodium dichloroisocyanurate is known to undergo heat-induced decomposition. In order to prevent such decomposition, which can result in actual fires, careful monitoring of the process is required. Additionally, it is essential to avoid contamination of the product with organics such as lubricating oil, since this can cause thermal decomposition of the product.

I have discovered that the granulation of powdery (−200 mesh) sodium dichloroisocyanurate can be safely and efficiently carried out by using a sheeting compactor with sheeting rolls which operate at conventional pressures, of the order of $7,500 \pm 50\%$ pounds of force per linear inch of roll, and feeding to the rolls powdered sodium dichloroisocyanurate which contains from about 11.0 to about 14.0% of water of hydration. This feed produces a sheet which is broken up into a major proportion of +70 mesh material in a standard granulator, reducing the recycle to under 40%, as compared with 60 to 80% when using anhydrous feed. The granular product of desired size, which is screened from the mix, may be used as such, or be dehydrated.

The drawing shows schematically the apparatus used in the practice of this invention.

The invention herein involves the sheeting of hydrated powdered sodium dichloroisocyanurate, and the breaking-up of the sheets into granules.

The process involves apparatus hitherto used in the granulation of anhydrous sodium dichloroisocyanurate; the apparatus is shown in the drawing.

Referring to the drawing, the powdered salt is fed into a hopper 10, and by means of feed screws 12 and 14 into the bite between a fixed roll 16 and a compression

roll 18, which is forced against the fixed roll 16 by a compression assembly 20. The sheet of salt 22 which results is fed into a granulator 24 which breaks up the sheet into granules and powder; the product is removed at 26, and is then screened.

The roll 18 may be smooth or otherwise designed, as in the form of a sine wave, to produce the particle size desired for the granulated sodium dichloroisocyanurate product. A typical 4 inch sine wave which has about ten complete $\frac{3}{8}$ inch sine waves in it, about $\frac{1}{4}$ inch deep, produces a corrugated sheet which breaks up into mainly −10 + 70 product.

The roll 18 is forced against the roll 16 by about $7,500 \pm 50\%$ pounds of force per linear inch of roll. The granulator 24 may be any device which will break up the continuous sheet which is fed from the roller. Since the sheet is readily friable, a simple hammer mill will work, or a mill with simple rotating shear blades (e.g. a Fitz mill).

In operation with powdered anhydrous feed, as heretofore practiced, rather high recycle rates are obtained. In typical runs, recycle of fines runs from 60%, under the very best circumstances, to about 80%, which is the normal operating result.

The examples given herein are by way of example only and not by way of limitation. All percents are by weight unless otherwise stated.

EXAMPLE A — ANHYDROUS FEED (COMPARATIVE EXAMPLE)

In a typical run with anhydrous feed a plant-produced sodium dichloroisocyanurate powder, predominantly −230 +400 U.S. mesh and containing 0 to 5% of moisture was fed at 7,500 pounds of force per linear inch of roll into the compactor described above, operated at a roll speed of 15 rpm, a vertical-screw speed of 840 rpm, and a horizontal-screw speed of 1,300 rpm. The corrugated sheet produced was fed into the granulator. In continuous processing at the rate of about 1,800 pounds per hour, handling thousands of pounds of material, the following particle size distribution was obtained:

U.S. Screen Size	Weight Percent	
−10 + 20	2.0	
−20 + 30	6.0	
−30 + 70	14.0	
−70 + 100	8.0	} Recycle
−100	70.0	

Note the recycle rate of 78%, which is typical of ordinary production. Under best circumstances, the recycle rate is 60%; it generally runs nearer to 80%.

Additionally, it was necessary to keep a careful eye on the machine, to prevent fires.

EXAMPLE 1

The same compactor rolls and granulator, at the same settings as in Example A were fed with the same raw material, first hydrated with 14.1% of water (the dihydrate); the material had the same particle size distribution as the original material. The product resulting had the following particle size distribution:

U.S. Screen Size	Weight Percent	
- 10 + 20	38.9	
- 20 + 30	8.7	
- 30 + 70	14.5	
- 70 + 100	3.2	Recycle
-100	34.7	

Note the reduction in recycle rate, with the resultant startling increase in production possible.

EXAMPLE 2

Example 1 was repeated, except the feed contained 12.6% of water, and the rolls were set at a force of about 8,000 pounds per linear inch of roll. The results were as follows:

U.S. Screen Size	Weight Percent	
+ 10	1.1	— Recycle
- 10 + 20	33.4	
- 20 + 30	8.6	
- 30 + 70	14.6	
- 70 + 100	3.0	Recycle
-100	39.2	

It was noted that small amounts of free water (e.g. 14.7% total = 0.6% of free water) further reduced the recycle rate (to below 20% total of material of -70

mesh size in one experiment), but an irritating odor developed during the run, indicating some decomposition of the material. Hence, free water should be avoided.

I use at least about 11% of water, this being the point at which the product will no longer sustain propagation of thermal decomposition. At this figure, recycle rates are somewhat higher than when fully hydrated material (14.1% of water) is used, but the recycle rate is still sharply lower than with anhydrous material.

As indicated above, the product can be used as it comes from the process, or it can be dehydrated to produce the anhydrous product.

Obviously, the examples can be multiplied without departing from the scope of the invention as defined in the claim.

What is claimed is:

1. In the method of producing granular sodium dichloroisocyanurate from -200 mesh powder in which the powder is fed between two rolls, and one of the rolls is forced against the other with a force of 7,500 ± 50% pounds per linear inch, to produce a continuous sheet of sodium dichloroisocyanurate which is then broken up into granules and sized, the improvement which comprises feeding the powdered sodium dichloroisocyanurate as hydrated material containing 11 to 14% of water of hydration, and free of water not bound as water of hydration whereby the recycle of fines in the process is reduced from a major percentage to a minor percentage.

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