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(54) **METHODE POUR AMELIORER LA FLUIDITE DU CHLORURE
DE CYANURYLE SOLIDE**

(54) **PROCESS FOR IMPROVEMENT OF THE FLOWABILITY OF
SOLID CYANURIC CHLORIDE**

(57) Solid cyanuric chloride obtained by desublimation or prilling is subjected to a shearing stress in a kneader or mixer while heating to a temperature below the melting point so as to improve the flowability of the cyanuric chloride.



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ABSTRACT

Solid cyanuric chloride obtained by desublimation or prilling is subjected to a shearing stress in a kneader or mixer while heating to a temperature below the melting point so as to improve the flowability of the cyanuric chloride.

PROCESS FOR IMPROVEMENT OF THE FLOWABILITY OF
SOLID CYANURIC CHLORIDE

5 This invention relates to a process for improvement of the flowability of solid cyanuric chloride.

10 Cyanuric chloride is an important intermediate product in the production of dyes, optical brighteners, plant protective agents and pharmaceutical active ingredients as well as textiles, paper and plastic auxiliary agents.

15 Commercially, cyanuric chloride is produced by trimerization of cyanogen chloride in the gaseous phase. The gaseous cyanuric chloride resulting from the trimerization is either directly desublimated by introduction into a cold inert gas stream or first liquified and then prilled. In this case, it is obtained in the form of a fine-particle powder. It is known that this powder, especially the powder obtained by desublimation, has only an unsatisfactory flowability.

20 This poor flowability leads to difficulties in storage, conveying, feeding and further processing of the product. During storage, it tends to lump and is subject to bridge formation, which makes discharge from a silo more difficult. In conveying by means of pipelines blockage easily occurs. Feeding and, for example, dissolving in solvents in further processing are hindered by uneven material flow. Therefore, it is necessary and customary to take steps to improve the flowability of the cyanuric chloride. For this purpose, highly dispersed silicic acids, which are known, e.g., under the designation Aerosil®, are added to the product and also are used in numerous other materials as flow auxiliary agents. These substances are indeed largely inert, but for certain uses it is undesirable to mix foreign substances with the cyanuric chloride.

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An object of the invention is to provide a process which without great industrial outlay and especially without the use of troublesome additives, improves the flow characteristics of the fine-particle cyanuric chloride powder while retaining its reactivity.

Accordingly, the invention provides a process for improving the flowability of solid cyanuric chloride, which comprises subjecting cyanuric chloride powder to a particle agglomeration in a kneader or mixer without the addition of binders at a temperature of from 20° to 146°C under the influence of shearing.

It was surprisingly found that cyanuric chloride powder, which was stirred or kneaded at elevated temperature and under the action of sufficiently great shear forces, changes its grain size distribution and, in particular, considerably improves its flow characteristics. It is known in the art that such treatment can lead to a particle enlargement. Thus, for example, the use of mixers of various types for agglomeration of fine-particle materials is described by C.E. Capes in Kirk-Othmer's Encyclopedia of Chemical Technology, 3rd edition. Vol. 21, pp. 77 to 87. However, in these known treatments the presence of a liquid phase, either in the form of a sprayed-on binder or in the form of moisture of the very material to be agglomerated, is necessary to obtain solid agglomerates, which do not immediately decompose again with mechanical stress.

In contrast, the process according to the invention succeeds without the addition of liquid or binder and yet leads to compression-resistant grains of cyanuric chloride.

The cyanuric chloride may be circulated, according to the process of the invention, either directly after removal from the desublimation chamber or the prilling chamber or after intermediate storage, in a kneader or mixer at a temperature of from 20° to 146°C, preferably 60°

to 120°C. The kneading or mixing device is preferably heatable. Especially preferred are heatable mixers with rotating mixing rotors, such as plowshare, vane or paddle mixers. The heat input can take place both by outside heating, for example in the form of a heating jacket through which flows one of the usual heat transfer media or which contains electric heating elements, or by appropriately designed kneading or mixing rotors or by a combination of the two methods.

It is also possible to produce the heat input entirely or partially by blowing a heated inert gas into the kneader or mixer or the cyanuric chloride contained in it. In this case, the waste gases which inevitably contain some evaporated cyanuric chloride, advantageously are recycled into the desublimation chamber to keep the substance losses as small as possible. An especially fast and efficient heat input can be achieved by a combination of the described methods.

The period of the treatment is preferably between 10 minutes and 10 hours and depends on the temperature, the intensity of the mixing process and the desired grain size distribution. Under the conditions selected in the following preparatory examples, treatment periods of 1 to 2 hours have proved to be especially advantageous.

Besides the thermal action, the shear forces which occur play a role in achieving the grain structure. The speed of the kneading or mixing elements, therefore, is suitably selected so that a sufficient shear stress of the material to be treated results from the peripheral speed and the dimensions of the shear gaps. The peripheral speed of the kneading or mixing elements is preferably between 0.1 and 10 m/s, especially preferred being the range of 2 to 5 m/s.

After treatment, the cyanuric chloride is advantageously cooled first to a temperature in the range of 10° to 50°C, preferably from 20° to 30°C, i.e. about to

room temperature. The cooling can take place in the same device as the mechanical treatment, but it is also possible to perform the cooling in a separate mixer of similar or different design. Since in this case only an adequate heat transfer has to be guaranteed, for this purpose devices can also be used in which the charge is not subject to any substantial shear stress, such as, drums or tumbling mixers. The heat removal can take place in the same way as the heat input by means of contact with the walls and/or the mixing elements, but it is also possible to achieve a direct cooling by introduction of cold or condensed gas, such as air or nitrogen in liquid or gaseous state or liquefied carbon dioxide.

After cooling, the cyanuric chloride is discharged in a known way and can then be packaged or stored. If so desired, a further improvement of the flowability can be achieved by mixing the cyanuric chloride with a known auxiliary agent, such as the above-mentioned Aerosil®.

The following examples illustrate the invention.

Examples 1 to 3 were performed with desublimated cyanuric chloride. The flowability was determined in each case by the Jenike shear test. The ratio of the bulk-solidifying stress σ_1 to the bulk solidity f_c is considered as the measurement for the flowability. This ratio is identified as Jenike flow function FF_c ($FF_c = \sigma_1/f_c$). The untreated cyanuric chloride typically exhibits the following values:

Without free-flow agent addition: $FF_c = 1.8-2.0$

With free-flow agent addition: $FF_c = 4.0-6.0$

EXAMPLE 1

1 kg of cyanuric chloride with an average grain size of 11 microns was kneaded in a heatable kneader with 2-liter capacity at 100°C with 40 rpm (peripheral speed = 0.12 m/s). As early as one hour from commencement, the average grain size had increased to 15 microns and the FF_c to 14. With continuation of the treatment the average

grain size constantly increased and after a total of 6 hours reached a value of 40 microns.

EXAMPLE 2

5 In a heatable plowshare mixer (brand LOEDIGE, model VT50) 25 kg of cyanuric chloride was treated at a temperature of 85°C and a peripheral speed of the mixing elements of 2.5 m/s. The average grain size increased from an initial value of 11 microns to 15 microns after 1 hour and to 25 microns after 2 hours. The flowability was
10 already considerably improved after one hour, so that, following addition of a free-flow agent, an FF_c value of 21 could be measured.

EXAMPLE 3

15 A similar procedure as in Example 2 was followed, but with a 95°C temperature of the mixture contents and a peripheral speed of 3.14 m/s. The average grain size increased from 11 microns to 18 microns (after 1 hour) and 25 microns (after 2 hours). The FF_c value was 5.5 (without free-flow agent addition) or 9.7 (with free-flow agent
20 addition).

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A process for improving the flowability of solid cyanuric chloride, which comprises subjecting cyanuric chloride powder to a particle agglomeration in a kneader or mixer without the addition of a binder at a temperature in the range of about 20° to 146°C under the influence of shearing.

2. A process according to claim 1, wherein the particle agglomeration takes place at a temperature of from 60° to 120°C and the product is then cooled to room temperature.

3. A process according to claim 2, wherein the cooling takes place indirectly by means of contact with the mixing elements and/or the wall of the kneader or mixer.

4. A process according to claim 2, wherein the cooling takes place directly by the introduction of an inert cooling medium.

5. A process according to claim 1, 2, 3 or 4, wherein the heat input necessary to reach the mixing temperature takes place wholly or partially by means of the mixing elements and/or the wall of the kneader or mixer.

6. A process according to claim 1, 2, 3 or 4, wherein the heat input necessary to reach the mixing temperature takes place wholly or partially by blowing a hot inert gas into the kneader or mixer.

7. A process according to claim 1, 2, 3 or 4 wherein the peripheral speed of the kneading or mixing elements is from 0.1 to 10 m/s.

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8. A process according to claim 1, 2, 3 or 4, wherein the peripheral speed of the kneading or mixing elements is from 2 to 5 m/s.

9. A process according to claim 1, 2, 3 or 4, wherein the retention time of the cyanuric chloride in the mixer or kneader is from 10 minutes to 10 hours.

10. A process according to claim 1, 2, 3 or 4, wherein the retention time of the cyanuric chloride in the kneader or mixer is from 1 to 2 hours.

11. A process according to claim 1, wherein the particle agglomeration is performed in a heatable plowshare mixer.

12. A process according to claim 11, wherein the flowability is further improved by admixing a highly dispersed silicic acid in with the cyanuric chloride.