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(54) **Titre** : GRANULAT CONSTITUE DE PERCARBONATE DE SODIUM EN LIT FLUIDISE ET PROCEDE DE FABRICATION
(54) **Title**: SODIUM PERCARBONATE FLUID BED GRANULATED MATERIAL AND METHOD FOR THE PRODUCTION THEREOF

(57) **Abrégé/Abstract**:

The invention relates to a method for producing granular sodium percarbonate with a low TAM value by means of fluid bed granulation. According to the invention, during the fluid bed granulation, a magnesium compound in a quantity of 50 to 2000 ppm, especially 100 to 1000 ppm, and/or a complexing agent from the series of hydroxycarboxylic acids, aminophosphonic acids, phosphonocarboxylic acids, and hydroxyphosphonic acids, and the alkali salts, ammonium salts or magnesium salts thereof in a quantity of 50 to 2000 ppm, especially 200 to 1000 ppm, are added to the soda and/or H_2O_2 solution as stabilisers. Preferably, a combination of a magnesium compound in a quantity of 100 to 1000 ppm Mg^{2+} and water glass in a quantity of 0.1 to 1 wt. %, especially 0.1 to 0.5 wt. %, is used. In this way, it is possible to obtain granulated material having a TAM value of 6 μW/g or below and a low dissolution time.

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(54) Title: SODIUM PERCARBONATE FLUID BED GRANULATED MATERIAL AND METHOD FOR THE PRODUCTION THEREOF

(54) Bezeichnung: NATRIUMPERCARBONAT-WIRBELSCHICHTGRANULAT UND VERFAHREN ZU SEINER HERSTELLUNG

(57) Abstract: The invention relates to a method for producing granular sodium percarbonate with a low TAM value by means of fluid bed granulation. According to the invention, during the fluid bed granulation, a magnesium compound in a quantity of 50 to 2000 ppm, especially 100 to 1000 ppm, and/or a complexing agent from the series of hydroxycarboxylic acids, aminophosphonic acids, phosphonocarboxylic acids, and hydroxyphosphonic acids, and the alkali salts, ammonium salts or magnesium salts thereof in a quantity of 50 to 2000 ppm, especially 200 to 1000 ppm, are added to the soda and/or H_2O_2 solution as stabilisers. Preferably, a combination of a magnesium compound in a quantity of 100 to 1000 ppm Mg^{2+} and water glass in a quantity of 0.1 to 1 wt. %, especially 0.1 to 0.5 wt. %, is used. In this way, it is possible to obtain granulated material having a TAM value of 6 µW/g or below and a low dissolution time.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren zur Herstellung von granulatförmigem Natriumpercarbonat durch Wirbelschicht-Sprühgranulation mit niedrigem TAM-Wert. Erfindungsgemäß werden bei der Wirbelschicht-Sprühgranulation eine Mg-Verbindung in einer Menge von 50 bis 2.000 ppm, insbesondere 100 bis 1.000 ppm, oder/und ein Komplexbildner aus der Reihe der Hydroxycarbonsäuren, Aminocarbonsäuren, Aminophosphonsäuren und Phosphonocarbonsäuren, Hydroxyphosphonsäuren und deren Alkali-, Ammonium- oder Mg-Salze in einer Menge von 50 bis 2.000 ppm, insbesondere 200 bis 1.000 ppm, der Soda- und/oder H_2O_2-Lösung als Stabilisator zugesetzt. Vorzugsweise wird eine Kombination aus einer Mg-Verbindung in einer Menge von 100 bis 1.000 ppm Mg^{2+} und Wassersglas in einer Menge von 0,1 bis 1 Gew.-%, insbesondere 0,1 bis 0,5 Gew.-%, eingesetzt, und hierbei sind Granulate mit einem TAM-Wert um/unter 6 µW/g bei gleichzeitig geringer Lösezeit erhältlich.

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Sodium Percarbonate Fluid Bed Granulated Material And Method For The Production Thereof

Description

This invention relates to granular sodium percarbonate having a low TAM value (microcalorimetric determination of the energy release during storage, determined by means of TAM® Thermal Activity Monitor by Thermometric AB, Järfälla (SE)), in particular a TAM value of below 8 µW/g, wherein the grain of sodium percarbonate has a structure of the kind which is obtainable by a fluid-bed spray granulation and one or more stabilisers are substantially evenly distributed in the grain. The invention also relates to a process for producing the granular sodium percarbonate according to the invention, wherein an aqueous hydrogen peroxide solution and aqueous soda solution or soda suspension together with one or more stabilisers are sprayed into a fluid bed formed from sodium percarbonate particles and at the same time water is evaporated out of the fluid bed.

Crystallisation processes and so-called fluid-bed spray granulation processes in particular are used on a large scale for the production of sodium percarbonate corresponding to the general formula 2 $Na_2CO_3 \cdot 3 H_2O_2$, which is used as the bleaching component in detergents and cleaning agents. In the crystallisation processes - reference may be made, for example, to US Patent 4,146,571 - hydrogen peroxide and soda are reacted in a mother liquor containing common salt. To control the crystallisation and improve the stability of the sodium percarbonate, magnesium ions in a quantity of 0.2 to 2 wt.% are introduced at the same time as the hydrogen peroxide solution. 0.5 to 3 wt.% sodium hexametaphosphate is also added to the system. The sodium percarbonate thus obtained per se can be stored satisfactorily and has a low TAM value, but the active oxygen stability of the crystallisate in the presence of constituents of detergents, such as zeolites in particular, is unsatisfactory owing to the irregular surface and the not very dense structure. Sodium percarbonate which has been produced by a crystallisation process can be made more stable in storage in the presence of constituents of detergents by coating the grain of sodium percarbonate with stabilising components, such as sodium sulfate and other hydrate-forming substances, but the demands which are nowadays placed on such a product are frequently no longer adequately met.

In the crystallisation process for producing sodium percarbonate described in DE-PS 26 44 147, waterglass is used concomitantly in addition to a magnesium salt and sodium hexametaphosphate. The impurities in the mother liquor which result from the crystallisation are adsorbed onto the magnesium silicate during the precipitation of the latter and are separated off by filtration of the mother liquor prior to its reuse. However, the above-mentioned disadvantages still remain.

The fluid-bed spray granulation process yields substantially spherical, dense sodium percarbonate particles which have a shell-like structure owing to the production conditions and a greater stability in storage than that of a product obtained by crystallisation. The process is carried out by spraying an aqueous hydrogen peroxide solution and an aqueous soda solution or optionally soda suspension into a fluid bed containing sodium percarbonate particles whose diameters are smaller than those of the particles being produced. During the spraying in of the coreactants in aqueous medium, water is evaporated off at a fluid-bed temperature within the range of 40 to 95 °C.

In the fluid-bed spray granulation process according to DE-OS 27 33 935, the hydrogen peroxide solution and soda solution are mixed immediately prior to being sprayed. A condensed alkali metal phosphate or ammonium phosphate has to be added in order to avoid a premature crystallisation inside the nozzle. In addition, stabilisers such as magnesium sulfate may be added to the hydrogen peroxide solution and/or sodium silicate to the sodium carbonate solution. However, the addition of the above-mentioned stabilisers is not considered to be essential. The quantity of magnesium sulfate given in the Example results in a magnesium content of more than 1 g per kg of sodium percarbonate.

In the fluid-bed spray granulation process for producing granular sodium percarbonate according to DE-PS 43 29 205, the requirement to introduce a condensed phosphate is avoided by using a specially shaped three-component nozzle with external mixing of the solutions containing the coreactants. Again in this process, stability-enhancing additives, such as magnesium salts, waterglass, complexing agents, stannates and dipicolinic acid may be added to the solutions to be sprayed. No idea of the quantity of additives required, of the selection of the type of complexing agents nor of possible combinations of different stabilisers can be inferred from this document.

For reasons of safety in handling, in particular an increased safety during the storage of sodium percarbonate in a silo, there is nowadays an increasing demand for sodium percarbonate having a further improved storability, corresponding to a decreased TAM value compared with the prevailing values. The TAM value of sodium percarbonate can to a certain extent be improved, that is, lowered, by coating the sodium percarbonate with a material which renders it inert, but the effect thereby achievable is in many cases still inadequate. In the process according to

DE-OS 27 33 935, a low TAM value is obtained using the quantity of a condensed phosphate and of magnesium sulfate given in the Example, but the quantity of stabilisers required is very high.

Accordingly, the object of the present invention is to demonstrate another process for producing granular sodium percarbonate by fluid-bed spray granulation, whereby granular sodium percarbonate having as low a TAM value as possible can be obtained using as low a quantity as possible of one or more stabilisers. A further object is that, at the best possible stabilisation and hence low TAM value, the rate of dissolution of the sodium percarbonate should at the same time not be decreased, but rather increased.

These and other objects, which may be derived from the description below, are achieved by the process according to the invention.

Accordingly, a process for producing granular sodium percarbonate by fluid-bed spray granulation has been found, wherein an aqueous sodium carbonate solution or sodium carbonate suspension and an aqueous hydrogen peroxide solution in the presence of at least one stability-enhancing additive is sprayed into a fluid bed containing sodium percarbonate particles and at the same time water is evaporated at a fluid-bed temperature within the range of 40 to 95 °C, which is characterised in that the additive used is a magnesium compound in a quantity of 50 to 2,000 ppm Mg^{2+} and/or a chelate complexing agent from among the hydroxycarboxylic acids, aminocarboxylic acids, aminophosphonic acids, phosphonocarboxylic acids, hydroxyphosphonic acids and the alkali metal salts, ammonium salts or magnesium salts of the above-mentioned acids, in a quantity of 50 to 2,000 ppm, the quantity used in each case being based on the sodium percarbonate to be produced and a combination of a magnesium salt and a condensed phosphate being excepted. The subclaims are directed to preferred embodiments of the process according to the invention.

Of the magnesium compounds used as stabilisers, water-soluble compounds are preferred, for example, magnesium sulfate, magnesium chloride and magnesium acetate. It is particularly advantageous to employ magnesium salts of chelate complexing agents from among the hydroxycarboxylic acids, aminocarboxylic acids, aminophosphonic acids, phosphonocarboxylic acids and hydroxyphosphonic acids. Because they contain both magnesium and complexing agent, such salts act synergistically. In a preferred embodiment, magnesium compounds are used in a quantity corresponding to 100 to 1000 ppm (ppm = parts per million) Mg^{2+} and particularly preferably 100 to 500 and in particular 200 to 250 ppm Mg^{2+}. In the carrying out of the process, it is useful to add the magnesium compound to the hydrogen peroxide solution to be sprayed.

The chelate complexing agents used according to the invention are compounds selected from among nitrilotriacetic acid, iminodiacetic acid, ethylenediaminetetraacetic acid, iminodisuccinate, tartaric acid, gluconic acid, aminotri(methylene)phosphonic acid, ethylenediaminetetra(methylene)phosphonic acid, diethylenetriaminepenta(methylene)phosphonic acid, tri-, tetra-, penta- and hexamethylenetetra(methylene)phosphonic acid and 1-hydroxyethane-1,1-diphosphonic acid and their water-soluble salts, in particular sodium salts, ammonium salts and magnesium salts. The quantity of the chelate complexing agents used is preferably within the range of 100 to 1,000 ppm, in particular 200 to 1,000 ppm. In another preferred embodiment, the chelate complexing agents or their salts are added to the soda solution or soda suspension to be sprayed.

For the purpose of stabilisation, it is preferable also to add waterglass to the soda solution or soda suspension to be sprayed. Waterglass is generally used in a quantity of about 1 wt.% SiO_2, based on sodium percarbonate. A disadvantage of using too large a quantity of waterglass is that the rate of dissolution of the sodium percarbonate decreases. The experts are interested in lowering the SiO_2 content to values of significantly below 1 wt.%, based on sodium percarbonate, in order to minimise this disadvantage. By using one or more of the stabilising additives to be employed according to the invention and in addition waterglass, it is possible to lower the SiO_2 content to values of about or below 0.5 wt.% SiO_2, even 0.1 wt.% SiO_2.

The steps for carrying out the fluid-bed spray granulation process for producing sodium percarbonate are known to the experts - by way of example, reference is made to DE-OS 27 33 935 and DE-OS 43 29 205, whose disclosures are included in the present Description.

In a preferred embodiment of the process, a 30 to 75 wt.% hydrogen peroxide solution and a 20 to 60 wt.%, preferably 30 to 50 wt.% soda solution or soda suspension is sprayed into a fluid bed containing sodium percarbonate particles. At least one of these solutions or the suspension contains an effective concentration of at least one stabilising additive to be used according to the invention; preferably a synergistically effective combination of stabilisers is used. Particularly preferably the two solutions, or solution and suspension, are sprayed into the fluid bed using a three-component or four-component atomising nozzle with external mixing of the solutions. The above-mentioned nozzles comprise a central pipe and two or three jacketed pipes disposed around it, with the central pipe extending beyond the jacketed pipes by 2 to 10 times the radius of the central pipe. In the case of the three-component nozzle, the H_2O_2 solution is preferably passed through the central pipe and the soda solution or soda suspension through an adjacent jacketed pipe; a spraying gas for spraying the solutions is led through the outer jacketed pipe. In the case of the embodiment using a four-component atomising nozzle, spraying gas is preferably led through the jacketed pipe adjacent to the central pipe as well as through the outermost jacketed pipe; the two solutions are mixed externally as a result of this arrangement and at the same time a microclimate is produced. In view of the limited stability of sodium percarbonate, it is advisable to maintain the temperature of the fluid bed within the range of 50 to 70 °C during the spraying of the two solutions or of the H_2O_2 solution and soda suspension.

In a preferred embodiment, the fluid-bed spray granulation is carried out in a continuously operated fluid-bed apparatus with nucleation in the reactor or introduction of nuclei and classifying discharge of the granular material.

To increase the stability in storage of sodium percarbonate in the presence of constituents of detergents, such as zeolites in particular, it is advisable to coat the granular sodium percarbonate obtainable according to the invention with one or more layers subsequent to its formation. Here one can use the processes for coating sodium percarbonate known to the experts. The coating components are predominantly compounds which can themselves form hydrates, such as sodium sulfate, magnesium sulfate, sodium carbonate and sodium bicarbonate, borates and perborates. In many cases waterglass is additionally used as a component in a coating containing several components. In a particularly preferred embodiment, the coating consists substantially of sodium sulfate and its hydrates.

The invention also provides granular sodium percarbonate obtainable by the process according to the invention. The sodium percarbonate particles are substantially spherical and have a layered grain structure, as is typical for a fluid-bed spray granulation. The product contains, substantially evenly distributed in the grain, magnesium salt in a quantity within the range of 50 to 2,000 ppm Mg^{2+}, preferably 100 to 1000 ppm and in particular 200 to 500 ppm Mg^{2+}, and/or one or more chelate complexing agents, of the type previously disclosed, in a quantity of 50 to 2,000 ppm, in particular 200 to 1,000 ppm, but not a combination of a magnesium salt and a condensed phosphate. The products are further characterised in that they have a TAM value of equal to or less than 8 µW/g, in particular less than 7 µW/g, and particularly preferably about 4 to 6 µW/g. The given TAM values refer to a measurement by means of the Thermal Activity Monitor by Thermometric AB, Spjutvägen 5a, S-175 61 Järfälla; the measured values given are those found after storage in the measuring cuvette for 48 h at 40 °C. Preferably the product contains in addition waterglass having an SiO_2/Na_2O module of 1 to 3 in a quantity within the range of 0.1 to 0.5 wt.%. A combination of Mg^{2+} and SiO_2 enables low TAM values and, simultaneously, short dissolving times to be achieved even with a low input of SiO_2.

The advantages of the invention are that granular sodium percarbonate having a low TAM value can be obtained by a known fluid-bed spray granulation process through the use of stabilising additives and combinations of additives according to the invention. Owing to the low TAM value, it is possible to store sodium percarbonate in large quantities without creating a safety hazard. The selection of stabilising additives according to the invention additionally makes it possible to provide products which dissolve more rapidly than do many commercially available products having a silica content of about 1 wt.%. The following Examples illustrate the invention.

Examples

General instructions for procedure:

A 43 wt.% aqueous hydrogen peroxide solution and a 30 wt.% soda solution were sprayed by means of a three-component atomising nozzle into a laboratory apparatus for fluid-bed spray granulation, after fluidisation of sodium percarbonate placed therein. The apparatus was equipped with devices for dust recycling and introduction of nuclei as well as a classifying discharge. The spray rates were: 3.42 kg/h H_2O_2 solution and 9.54 kg/h soda solution. In each case magnesium sulfate was used as magnesium salt and added to the H_2O_2 solution. The chelate complexing agents used were added to the soda solution. Waterglass containing a module in the range of 1.8 to 2 (SiO_2/Na_2O) was added to the soda solution to be sprayed, in order to adjust the SiO_2 content of the sodium percarbonate. The tests using different stabilising additives and combinations of additives as well as different quantities of additives, in each case based on the sodium percarbonate to be prepared, and the resulting TAM values, determined after 48 h at 40 °C, may be seen in Tables 1 and 2 below. From these tests, it follows that the SiO_2 content can be decreased by the addition of magnesium sulfate, so that products which have a low TAM value - about 5 µW/g - and at the same time a high rate of dissolution are obtained.

As the tests represented in Table 3 show, the dissolving time of the sodium percarbonate decreases surprisingly sharply with an increasing quantity of Mg^{2+} at a constant, low SiO_2 content (here 0.1 wt.%).

Through the combination of magnesium with a chelate complexing agent, not only are products having a low TAM value obtained, but operational disturbances owing to precipitation of insoluble magnesium salts are at the same time avoided.

Whereas the use of chelate complexing agents according to the invention leads to a lowering of the TAM value, other known stabilisers for active oxygen compounds, such as dipicolinic acid, have proved to be scarcely effective as regards a lowering of the TAM value.

Determination of the dissolving time:- The dissolving time is the time in which 95 % of 2 g of the sodium perborate sample used is dissolved per l of water at 15 °C. This time is measured from the change in the electrical conductivity. A thermostatted glass measuring cell (D: = 90 mm, H_1 = 150 mm) is used for the determination. During the determination, the solution is stirred using a 4-blade stirrer at 340 ± 5 revolutions per minute.

Table 1: TAM values (40 °C / 48 h) in relation to the quantity(ppm) of Mg^{2+} and/or SiO_2

Example No.	Mg^{2+} (ppm)	SiO_2 (%)	Start O_a (%)	TAM 40 °C/48 h
1 **)	-	0.5	14.1	9.3
2 **)	25	0.5	13.9	8.4
3 **)	-	1.0	14.0	7.6
4	250	0.25	13.9	7.2
5	250	0.5	14.2	5.7
6	250	0.88	14.0	4.9
7	1000	0.5	13.6	3.4
8	480	0.25	13.69	5.7
9	Product of Example 6 coated with 5 wt.% Na_2SO_4		13.3	4.2

**) = not according to the invention

Table 2: TAM values (40 °C / 48 h) in relation to the type and quantity (ppm) of stabilising additives

Example No.	Chelate complexing agent *); quantity (ppm)	SiO_2 (%)	Start O_a (%)	TAM 40 °C/48 h
10	ATMP; 1000	0.5	14.2	5.3
11	HEDP; 1000	0.5	13.9	7.3
12	ATMP-Na_5; 1000	0.5	13.85	5.3
13	IDS; 1000	0.5	13.84	6.3
14	Na_7-DTPMP; 1000	0.5	13.83	6.1
15	DTPMP; 1000	0.5	13.99	5.0
16	EDPTP; 1000	0.5	13.96	5.1
17	EDTA; 1000	0.5	13.64	5.3
18	Gluconic acid; 1000	0.5	13.98	7.2

*) ATMP = Aminotri(methylene)phosphonic acid
Na_5-ATMP = Penta-Na salt of ATMP
HEDP = 1-Hydroxyethane-1,1-diphosphonic acid
IDS = Iminodisuccinate
DTPMP = Diethylenetriaminepenta(methylene)phosphonic acid
Na_7-DTPMP = Hepta-Na salt of DTPMP
EPTMP = Ethylenediaminetetra(methylene)phosphonic acid
EDTA = Ethylenediaminetetraacetic acid

Table 3: TAM value and dissolving time in relation to the quantity of Mg^{2+} introduced at a given SiO_2 content

Example No.	Mg^{2+} (ppm)	SiO_2 (%)	TAM value	Dissolving time (min/g)
19	125	0.1	10.4	1.4
20	250	0.1	9.3	1.1
21	500	0.1	6.3	0.8

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A process for producing granular sodium percarbonate by fluidised-bed spray granulation, wherein an aqueous sodium carbonate solution or sodium carbonate suspension and an aqueous hydrogen peroxide solution, in the presence of at least one stability-enhancing additive, are sprayed into a fluidised bed containing sodium percarbonate particles and at the same time water is evaporated at a fluidised-bed temperature within the range of 40 to 95°C; and

wherein a magnesium compound in a quantity of 50 to 2000 ppm Mg^{2+} is used as an additive, and water-glass having an SiO_2/Na_2O module in the range of 1 to 3, in a quantity corresponding to 0.1 to 1 wt.% SiO_2, is used as a stabiliser, the respective quantity used being based on the sodium percarbonate to be produced and a combination with a condensed phosphate being excluded.

2. A process according to claim 1, wherein a water-soluble Mg compound is used in a quantity of 100 to 1000 ppm Mg^{2+}.

3. A process according to claim 1, wherein a water-soluble Mg compound is used in a quantity of 200 to 500 ppm Mg^{2+}.

4. A process according to claim 2 or 3, wherein the Mg compound is a Mg sulfate, Mg acetate or Mg salt of a chelate complexing agent.

5. A process according to any one of claims 1 to 4, wherein the Mg compound is added to the aqueous H_2O_2 solution to be sprayed.

6. A process according to any one of claims 1 to 5, wherein water-glass in a quantity of 0.1 to 0.5 wt.% SiO_2, based on sodium percarbonate, is used as the stabiliser.

7. A process according to any one of claims 1 to 6, wherein in addition a chelate complexing agent is used in a quantity of 50 to 2000 ppm, said chelate complex agent being a hydroxycarboxylic acid, a phosphonocarboxylic acid, a hydroxyphosphonic acid, a salt of said acids, said salt being an alkali metal salt, an ammonium salt or a magnesium salt.

8. A process according to claim 7, wherein said acid is nitrilotriacetic acid, iminodiacetic acid, iminodisuccinate, tartaric acid, gluconic acid or 1-hydroxyethane-1,1-diphosphonic acid, and said chelate complexing agent is introduced in a quantity of 100 to 1000 ppm based on the

sodium percarbonate to be produced, and is added to the H_2O_2 solution and/or soda solution or soda suspension.

9. A process according to claim 8, wherein said chelate complexing agent is introduced in a quantity of 200 to 1000 ppm based on the sodium percarbonate to be produced.

10. A process according to any one of claims 1 to 9, wherein a 30 to 75 wt.% aqueous H_2O_2 solution, and a soda solution or soda suspension having an Na_2CO_3 content within the range of 20 to 60 wt.%, the H_2O_2 solution containing a Mg compound and the soda solution or soda suspension containing water-glass, are sprayed using a three-component or four-component atomising nozzle with external mixing and comprising a central pipe and two or three jacketed pipes disposed around it, with the central pipe extending beyond the jacketed pipes by 2 to 10 times the radius of the central pipe.

11. A process according to claim 10, wherein the Na_2CO_3 content is 30 to 50 wt.%.

12. A granular sodium percarbonate, having:

(i) a grain structure produced by fluidised-bed spray granulation;

(ii) substantially evenly in the grain, a magnesium compound in a quantity of 50 to 2000 ppm Mg^{2+} and in addition water-glass having an SiO_2/Na_2O module in the range of 1 to 3, in a quantity within the range of 0.1 to 0.5 wt.% SiO_2;

(iii) absence of a condensed phosphate; and

(iv) a thermal activity monitor (TAM) value of equal to or less than 8 μW/g measured after 48 h at 40 °C.

13. A granular sodium percarbonate according to claim 12, wherein the TAM value is less than 7 μW/g.

14. A granular sodium percarbonate according to claim 12 or 13, further comprising a single-layer or multilayer stabilising coating comprising one or more hydrate-forming salts.