

Reactive composition based on sodium bicarbonate and process for its
production

TECHNICAL FIELD

The invention relates to a reactive composition based on sodium bicarbonate. It relates more particularly to a reactive composition which can be used to purify a flue gas comprising impurities, for example a flue gas produced by the incineration of waste or the combustion of fossil fuels for the production of electricity. The invention also relates to a process for the production of this reactive composition and to a process for the purification of flue gases using it.

TECHNICAL BACKGROUND

Incineration is a technology which is becoming essential for the removal of household or municipal waste. The incineration of household waste is accompanied by the formation of a flue gas generally comprising hydrogen chloride. It is essential to remove the hydrogen chloride from the flue gas before discharging the latter to the atmosphere.

During the combustion of coal, for example in order to produce electricity, flue gases are emitted comprising sulphur oxides as acidic impurities.

A known process for purifying a flue gas comprising acidic compounds consists in treating the flue gas with sodium bicarbonate, so as to neutralize hydrogen chloride or sulphur oxides and to form sodium chloride or sodium sulphates.

More particularly, a process has been provided in which sodium bicarbonate is injected in the powder form into the flue gas exiting from the incinerator and the flue gas thus treated is subsequently sent to a filter (Solvay & Cie, brochure TR 895/5c-B-1-1290). In this known process, the flue gas has a temperature of less than 260°C at the point of injection of the sodium bicarbonate. The latter is employed in the form of a graded powder obtained by grinding, 90 % by weight of which is in the form of particles with a diameter of less than 16 µm.

In practice, the sodium bicarbonate powder employed in this known process also comprises sodium carbonate.

WO 95/19835 discloses a reactive composition based on sodium bicarbonate which has a high bicarbonate content and which is provided in the

form of a powder formed of particles, the particles having a specific distribution of dimensions. This composition is remarkably effective but is expensive.

In order to be able to generalize the application of purification treatments to the flue gases produced by industrial processes, it is important in many cases to reduce the cost of the reactants used.

EP 0 858 429 describes a composition comprising at least 80 % of sodium bicarbonate, less than 20 % by weight of sodium carbonate, from 0.2 % to 0.7 % by weight of ammonia, expressed as ammonium ions, and 2 to 10 % by weight of water. This composition, obtained by heat treatment of crude bicarbonate from an ammonia-soda plant, however releases high amounts of ammonia (NH_3) when stored, in particular in closed atmospheres. This generates rapidly ammonia concentrations above toxicity thresholds that are detrimental to the health of people handling such composition.

The invention is targeted at providing a reactive composition based on sodium bicarbonate which can be used in treatments for the purification of flue gases, presenting high amount of ammonia compounds useful in flue gas mitigation of nitrogen oxides by catalytic conversion to nitrogen, though releasing less ammonia during storage and handling than known compositions of the prior art. Moreover, the invention relates also to a process that renders possible to produce this composition under advantageous economic conditions.

SUMMARY OF THE INVENTION

The disclosure consequently relates to a reactive composition comprising between 60 % and 98 %, preferably between 80 and 98 %, by weight of sodium bicarbonate, between 1 % and 40 %, preferably between 1 % and 12 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , characterized in that the composition comprises less than 1.0 %, preferably less than 0.9 %, more preferably less than 0.8 % by weight of water.

In one aspect, there is provided a reactive composition comprising between 60 % and 98 % by weight of sodium bicarbonate, between 1 % and 40 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , characterized in that the composition comprises less than 1.0 %, preferably less than 0.9 %, more preferably less than 0.8 % by weight of water measured by weight loss at a temperature of 25 °C placing about 3 g of the composition in a watch-glass and

in a dissicator in the presence of silicagel under vacuum at about 50 mbar absolute pressure, during 24 hours.

In another aspect, there is provided a reactive composition comprising between 80 % and 98 % by weight of sodium bicarbonate, between 1 % and 20 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , wherein the composition is in the form of particles having a diameter D_{90} of less than 100 μm and a diameter D_{50} of less than 75 μm , preferably a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm , measured by laser diffractometry, characterized in that the composition comprises less than 1.0 %, preferably less than 0.9 %, more preferably less than 0.8 % by weight of water measured by weight loss at a temperature of 25 °C placing about 3 g of the composition in a watch-glass and in a dissicator in the presence of silicagel under vacuum at about 50 mbar absolute pressure, during 24 hours.

In a further aspect, there is provided a process for the production of a composition as disclosed herein, according to which :

- particles resulting from crude bicarbonate particles from an ammonia-soda plant are introduced into a gas stream at a temperature of more than 30°C comprising air, in order to form a gas stream laden with particles;
- the gas stream laden with particles is introduced into a mill in order to form a gas stream comprising ground particles having a diameter D_{90} of less than 100 μm and a D_{50} of less than 75 μm , preferably a D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm measured by laser diffractometry; to obtain a composition as disclosed herein.

In yet another aspect, there is provided a process for the purification of a flue gas comprising acidic impurities, such as hydrogen chloride or sulphur oxides, according to which a reactive composition as disclosed herein is introduced into the flue gas, at a temperature of 100 to 600°C, and the flue gas is subsequently subjected to a filtration or a dedusting.

DETAILED DESCRIPTION OF THE INVENTION

Before the present formulations of the invention are described, it is to be understood that this invention is not limited to particular formulations described, since such formulations may, of course, vary. It is also to be understood that the

5 terminology used herein is not intended to be limiting, since the scope of the present invention will be limited only by the appended claims.

As used herein, the singular forms "a", "an", and "the" include both singular and plural referents unless the context clearly dictates otherwise. By way of example, "an additive" means one additive or more than one additives.

5 The terms "comprising", "comprises" and "comprised of" as used herein are synonymous with "including", "includes" or "containing", "contains", and are inclusive or open-ended and do not exclude additional, non-recited members, elements or method steps. It will be appreciated that the terms "comprising", "comprises" and "comprised of" as used herein comprise the terms "consisting of", "consists" and "consists of".

10 Throughout this application, the term "about" is used to indicate that a value includes the standard deviation of error for the device or method being employed to determine the value.

As used herein, the term "average" refers to number average unless indicated otherwise.

15 As used herein, the terms "% by weight", "wt %", "weight percentage", or "percentage by weight" are used interchangeably.

The recitation of numerical ranges by endpoints includes all integer numbers and, where appropriate, fractions subsumed within that range (e.g. 1 to 5 can include 1, 2, 3, 4 when referring to, for example, a number of
20 elements, and can also include 1.5, 2, 2.75 and 3.80, when referring to, for example, measurements). The recitation of end points also includes the end point values themselves (e.g. from 1.0 to 5.0 includes both 1.0 and 5.0). Any numerical range recited herein is intended to include all sub-ranges subsumed therein.

25 All references cited in the present specification are hereby incorporated by reference in their entirety. In particular, the teachings of all references herein specifically referred to are incorporated by reference.

Unless otherwise defined, all terms used in disclosing the invention, including technical and scientific terms, have the meaning as commonly
30 understood by one of ordinary skill in the art to which this invention belongs. By means of further guidance, term definitions are included to better appreciate the teaching of the present invention.

In the following passages, different alternatives, embodiments and variants of the invention are defined in more detail. Each alternative and embodiment so
35 defined may be combined with any other alternative and embodiment, and this for each variant unless clearly indicated to the contrary or clearly incompatible

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when the value range of a same parameter is disjointed. In particular, any feature indicated as being preferred or advantageous may be combined with any other feature or features indicated as being preferred or advantageous.

Furthermore, the particular features, structures or characteristics described
5 in present description may be combined in any suitable manner, as would be
apparent to a person skilled in the art from this disclosure, in one or more
embodiments. Furthermore, while some embodiments described herein include
some but not other features included in other embodiments, combinations of
features of different embodiments are meant to be within the scope of the
10 invention, and form different embodiments, as would be understood by those in
the art.

The invention can be defined by the features according to the following
items:

Item 1. Reactive composition comprising between 80% and 98% by weight
15 of sodium bicarbonate, between 1% and 12% by weight of sodium carbonate and
between 0.02% and 2.0% by weight of ammonia, expressed in the form of
ammonium ions NH_4^+ , characterized in that the composition comprises less than
1.0 %, preferably less than 0,9 %, more preferably less than 0,8% by weight of
water.

20 Item 2. Composition according to the preceding Item in the form of
particles having a diameter D_{90} of less than 50 μm and a diameter D_{50} of less
than 35 μm , preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of
less than 20 μm , more preferably a diameter D_{90} of less than 30 μm and a
diameter D_{50} of less than 15 μm , measured by laser diffractometry.

25 Item 3. Composition according to Items 1 or 2 comprising between 2% and
12% by weight of sodium carbonate.

Item 4. Composition according to one of the preceding Items, comprising
between 85% and 95% by weight of sodium bicarbonate.

30 Item 5. Composition according to one of the preceding Items, comprising
from 0.02% to 0.17% by weight of ammonia, expressed in the form of
ammonium ions NH_4^+ .

Item 6. Composition according to one of Items 1 to 4, comprising from
0.2% to 0.7% by weight of ammonia, expressed in the form of ammonium ions
 NH_4^+ .

Item 7. Composition according to one of Items 1 to 4, comprising more than 0.7% and less than 2.0% by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

5 Item 9. Composition according to one of the preceding Items, comprising from 0.01% to 5% by weight of additives selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, active charcoal, fatty acids and fatty acid salts.

10 Item 10. Process for the production of a composition according to one of the preceding Items, according to which:

- particles resulting from crude bicarbonate particles from an ammonia-soda plant are introduced into a gas stream at a temperature of more than 30°C comprising air, in order to form a gas stream laden with particles;
- the gas stream laden with particles is introduced into a mill in order to form
15 a gas stream comprising ground particles having a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm measured by laser diffractometry.

20 Item 11. Process according to the preceding Item, in which the crude bicarbonate particles from an ammonia-soda plant have a diameter D_{90} of greater than 100 μm and a diameter D_{50} of greater than 50 μm .

25 Item 12. Process according to one of Items 10 or 11, in which the crude bicarbonate particles from an ammonia-soda plant have a water content of at most 8% in weight, preferably of at most 6%, more preferably at most 5% in weight before being introduced into the gas stream.

Item 13. Process according to one of Items 10 to 12, in which the crude bicarbonate particles are mixed with other compounds, in particular with from 0,01 % to 5% in weight additives.

30 Item 14. Reactive composition according to one of Items 1 to 9, which can be obtained by the process according to one of Items 10 to 13.

Item 15. Process for the purification of a flue gas comprising acidic impurities, such as hydrogen chloride or sulphur oxides, according to which a reactive composition according to one of Items 1 to 9 or according to Item 14 is
35 introduced into the flue gas, at a temperature of 100 to 600°C, and the flue gas is subsequently subjected to a filtration or a dedusting.

Item 16. Process according to Item 15 wherein the flue gas subjected to a filtration is, subsequently the filtration, subjected to a selective catalytic reduction of nitrogen oxides (SCR DeNO_x).

Alternatively, the invention can be defined by the features according to the following items:

Item 1'. Reactive composition comprising between 60 % and 98 % by weight of sodium bicarbonate, between 1 % and 40 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , characterized in that the composition comprises less than 1.0 %, preferably less than 0,9 %, more preferably less than 0,8 % by weight of water.

Item 2'. Composition according to the preceding Item comprising between 80 and 98 %, by weight of sodium bicarbonate.

Item 3'. Composition according to Items 1' or 2' comprising between 1 % and 12 % by weight of sodium carbonate.

Item 4'. Composition according to Items 1' or 2' comprising between 2 % and 12 % by weight of sodium carbonate.

Item 5'. Composition according to one of Items 1' to 4', comprising between 85 % and 95 % by weight of sodium bicarbonate.

Item 6'. Composition according to one of Items 1' to 5' in the form of particles having a diameter D_{90} of less than 100 μm and a diameter D_{50} of less than 75 μm , preferably a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm , measured by laser diffractometry.

Item 7'. Composition according to one of Items 1' to 6', comprising from 0.02 % to 0.17 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

Item 8'. Composition according to one of Items 1' to 6', comprising from 0.2 % to 0.7 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

Item 9'. Composition according to one of Items 1' to 6', comprising more than 0.7 % and less than 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

Item 10'. Composition according to one of Items 1' to 9', comprising from 0.01 % to 5 % by weight of additives selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, activated carbon or active charcoal, fatty acids and fatty acid salts.

Item 11'. Process for the production of a composition according to one of Items 1' to 10', according to which:

- particles resulting from crude bicarbonate particles from an ammonia-soda plant are introduced into a gas stream at a temperature of more than 30°C comprising air, in order to form a gas stream laden with particles;
- the gas stream laden with particles is introduced into a mill in order to form a gas stream comprising ground particles having a diameter D_{90} of less than 100 μm and a D_{50} of less than 75 μm , preferably a D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm measured by laser diffractometry.

Item 12'. Process according to the preceding Item, in which the crude bicarbonate particles from an ammonia-soda plant have a diameter D_{90} of greater than 80 μm , preferably greater than 100 μm and a diameter D_{50} of greater than 50 μm .

Item 13'. Process according to one of Items 11' or 12', in which the crude bicarbonate particles from an ammonia-soda plant have a water content of at most 15 % in weight, preferably at most 12 %, more preferably at most 10 %, and even more preferably at most 8 % in weight of water before being introduced into the gas stream.

Item 14'. Process according to one of Items 11' to 13', in which the crude bicarbonate particles are mixed with other compounds, in particular with from 0.01 % to 5 % in weight additives.

Item 15'. Reactive composition according to one of Items 1' to 10', which can be obtained by the process according to one of Items 11' to 14'.

Item 16'. Process for the purification of a flue gas comprising acidic impurities, such as hydrogen chloride or sulphur oxides, according to which a reactive composition according to one of Items 1' to 10' or according to Item 15'

is introduced into the flue gas, at a temperature of 100 to 600°C, and the flue gas is subsequently subjected to a filtration or a dedusting.

Item 17'. Process according to Item 15' wherein the flue gas subjected to a filtration is, subsequently the filtration, subjected to a selective catalytic
5 reduction of nitrogen oxides (SCR DeNOx).

Further alternatively, the invention can be defined by the features according to the following items:

Item 1''. Reactive composition comprising between 60 % and 98 % by weight of sodium bicarbonate, between 1 % and 40 % by weight of sodium
10 carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , characterized in that the composition comprises less than 1.0 %, preferably less than 0,9 %, more preferably less than 0,8 % by weight of water.

Item 2''. Composition according to Item 1'' wherein the composition is in
15 the form of particles having a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , measured by laser diffractometry.

Item 3''. Reactive composition comprising between 80 % and 98 % by weight of sodium bicarbonate, between 1 % and 20 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in
20 the form of ammonium ions NH_4^+ , wherein the composition is in the form of particles having a diameter D_{90} of less than 100 μm and a diameter D_{50} of less than 75 μm , preferably a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less
25 than 30 μm and a diameter D_{50} of less than 15 μm , measured by laser diffractometry, characterized in that the composition comprises less than 1.0 %, preferably less than 0,9 %, more preferably less than 0,8 % by weight of water.

Item 4''. Composition according to one of Items 1'' or 2'' comprising between 80 and 98 %, by weight of sodium bicarbonate.

Item 5''. Composition according to one of Items 1'', 2'' or 4'' comprising between 1 % and 12 % by weight of sodium carbonate.

Item 6''. Composition according to one of Item 1'' to 5'' comprising between 2 % and 12 % by weight of sodium carbonate.

Item 7''. Composition according to one of Items 1'' to 6'', comprising
35 between 85 % and 95 % by weight of sodium bicarbonate.

Item 8". Composition according to one of Items 1" to 7", comprising from 0.02 % to 0.17 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

5 Item 9". Composition according to one of Items 1" to 7", comprising from 0.2 % to 0.7 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

Item 10". Composition according to one of Items 1" to 7", comprising more than 0.7 % and less than 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

10 Item 11". Composition according to one of Items 1" to 10", comprising from 0.01 % to 5 % by weight of additives selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, activated carbon or active charcoal, fatty acids
15 and fatty acid salts.

Item 12". Process for the production of a composition according to one of Items 1" to 11", according to which:

- particles resulting from crude bicarbonate particles from an ammonia-soda plant are introduced into a gas stream at a temperature of more than 30°C
20 comprising air, in order to form a gas stream laden with particles;
- the gas stream laden with particles is introduced into a mill in order to form a gas stream comprising ground particles having a diameter D_{90} of less than 100 μm and a D_{50} of less than 75 μm , preferably a D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than
25 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm measured by laser diffractometry.

Item 13". Process according to Item 12", in which the crude bicarbonate particles from an ammonia-soda plant have a water content of at most 15 % in
30 weight, preferably at most 12 %, more preferably at most 10 %, and even more preferably at most 8 % in weight of water before being introduced into the gas stream.

Item 14". Process for the purification of a flue gas comprising acidic impurities, such as hydrogen chloride or sulphur oxides, according to which a
35 reactive composition according to one of Items 1 to 11 is introduced into the flue

gas, at a temperature of 100 to 600°C, and the flue gas is subsequently subjected to a filtration or a dedusting.

Item 15". Process according to Item 14" wherein the flue gas subjected to a filtration is, subsequently the filtration, subjected to a selective catalytic
5 reduction of nitrogen oxides (SCR DeNOx).

Item 16". Composition according to one of Items 1" to 11" wherein the ammonia content, expressed in the form of ammonium ions NH_4^+ , is measured by distillation of an aqueous solution obtained by dissolving the sample in deionized water alkalinized with caustic soda at pH more than 11 (alkaline
10 distillation method; second alternative form below).

The inventors have observed, as a first advantage, that such a reactive composition presents a reduced release of ammonia when stored or handled compared to previously known compositions, though being still very effective for numerous applications, in particular for the treatment of flue gases, despite its
15 lower bicarbonate content compared to standard technical sodium bicarbonate.

In the invention, generally, the ammonia is defined as being gaseous ammonia (NH_3), adsorbed and absorbed in the particles based on sodium bicarbonate and the remaining moisture, as released, by heating at 30°C during two hours.

20 In a first alternative form, it is advantageous for the ammonia under consideration to also comprise the gaseous ammonia released by heating at 120°C during 2 hours, of the ammonium bicarbonate, ammonium carbonate, ammonium carbamate, sodium carbamate and other instable ammonium compounds at this temperature. In a second alternative form, which is the preferred
25 form (so-called alkaline distillation method), the ammonia comprises also the ammonia released by distillation of an aqueous solution obtained by dissolving the sample in deionized water alkalinized with caustic soda at pH more than 11, and enabling the measurement of ammonium species such as ammonium bicarbonate, ammonium carbonate, ammonium carbamate, sodium carbamate, ammonium
30 chloride, and other ammonium salts such as the ammonium salts contained in crude sodium bicarbonate obtained from the ammonia Solvay process. In a third alternative form, the ammonia comprises any ammonia-comprising entity. In this case, the total nitrogen, expressed in the form of ammonium ions, is measured. The three of these alternative forms can be applied to all the
35 embodiments described in this account, in which embodiments an ammonia content is specified.

The determination according to the invention and the first alternative form of the ammonia expressed in the form of ammonium ions NH_4^+ of a product sample is done by capturing the released gaseous ammonia (NH_3) from the heating operation by condensation in a scrubber with a HCl solution to transform the ammonia into NH_4^+ , which is analysed by a colorimeter.

The determination according to the second alternative form of the ammonia expressed in the form of ammonium ions NH_4^+ of a product sample is done by measurement with a spectro-colorimeter Docteur Lange X-500 on 150 mL of distillate from distilling a solution of 50 g of product, 150 mL of deionized water and 90 mL of a solution of caustic soda NaOH 9N. Cuvettes Dr Lange LCK 304, range 0.02 – 2.5 mg NH_4^+ /L are used.

The determination according to the third alternative form of the ammonia expressed in the form of ammonium ions NH_4^+ of a product sample is done by measurement by spectrophotometry of a solution sample by using a kit of Dr Lange LCK 338 Total Nitrogen, range 20 -100 mg/L. Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol. Alternatively the determination according to the third alternative form of the ammonia expressed in the form of ammonium ions NH_4^+ of a product sample is done by transforming all the ammonia species (NH_4Cl , NH_4HCO_3 , sodium carbamate, ammonium carbamate) into acidic form of NH_4^+ , and then potentiometric titration of NH_4^+ with NaClO in a bicarbonate assay medium according the following reaction :



For this determination 5 g of the product sample is dissolved into 50 ml of deionized water in presence of methyl orange as pH indicator (pH 3.1-4.4), acidified with H_2SO_4 2N down to orange colour change, then 50 ml of a saturated solution with sodium bicarbonate is added, and the point of equivalence when adding a NaClO solution of 0.2N, is determined by potentiometry.

In an advantageous embodiment of present invention, the composition is provided in the form of particles having a diameter D_{90} of less than 100 μm and a diameter D_{50} of less than 75 μm , preferably a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm , measured by laser diffractometry. When the particles of the composition have a

diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm , it is even more advantageous that the D_{50} to be less than 12 μm , preferably less than 10 μm .

Indeed, it has been observed in the applications for the treatment of flue gases, that the combination between the specific distribution of the diameters of the particles of the composition according to the invention, having a few large particles, and between the specific content of ammonia has appeared essential for its effectiveness. The ammonia content has a beneficial effect on the catalytic reduction of nitrogen oxides, on the height of the specific surface (m^2/g BET) of the bicarbonate particles after thermal transformation into sodium carbonate and thus on the efficiency of the sorbent. Without wishing to be committed to a theoretical explanation, the inventors believe that this second advantage of present reactive composition is due to the ammonia content closely related to the particle size distribution, with which it is in equilibrium.

Advantageously the reactive composition of the present invention comprises between 2 % and 12 %, preferably between 2% and 10% by weight of sodium carbonate.

It is recommended that the reactive composition comprises between 85 % and 95 % by weight of sodium bicarbonate.

In an advantageous embodiment, the reactive composition is provided in the form of particles having a particles size distribution slope σ of less than 2.

The particles size distribution slope σ is defined by :

$$\sigma = \frac{D_{90} - D_{10}}{D_{50}}$$

D_{90} , respectively D_{50} and D_{10} , represent the equivalent diameter for which 90 % (respectively 50 % and 10 %) of the weight of the particles of the reactive composition have a diameter of less than D_{90} (respectively D_{50} and D_{10}). These particle size parameters are defined by the laser ray diffraction analytical method.

Advantageously the reactive composition of present composition is a powder with a free flowing density of at least 0.4, preferably at least 0.45, more preferably at least 0.50 kg/dm^3 . Generally the free flowing density of the reactive composition of present composition is at most 0.8, or at most 0.7, or at most 0.6, or at most 0.55 kg/dm^3 , preferably of 0.45 to 0.55 kg/dm^3 .

In an alternative form of the reactive composition according to the invention, the composition comprises between 85 % and 95 % by weight of sodium bicarbonate.

According to a first variant of the invention, the composition comprises
5 between 0.02 % and 0.17 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ . This first variant is particularly advantageous for use of the composition in animal food, as ammonia release is particularly low in closed environment. Such compositions show also particularly low caking behaviour during storage in silos or big bags.

10 According to a second variant of the invention, the composition comprises from 0.2 % to 0.7 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ . This second variant is advantageous for providing larger ammonia amount in flue gas mitigation use, and though presenting a lower ammonia release at room temperature during storage compared to prior art.

15 According to a third variant of the invention, the composition comprises more than 0.7 % and less than 2.0 % by weight of ammonia, more advantageously more than 0.8 % and less than 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ . This second variant is advantageous for providing larger ammonia amount in flue gas mitigation use,
20 and is particularly advantageous with the above described specific distribution of the diameters of the particles of the composition, having a few large particles, showing an improved effect on the catalytic reduction of nitrogen oxides.

In the present invention, the reactive composition comprises less than 1 % by weight of water. Generally, the reactive composition comprises at most 0.9 %,
25 advantageously at most 0.8 %, more advantageously at most 0.7 %, even more advantageously at most 0.5 % by weight of water. There is no need for the present composition to have very low amount of water to provide a decreased release of ammonia during storage. Moreover a very low amount of water is detrimental to the quality of the reactive composition, as most of the techniques
30 of drying sodium bicarbonate, lead also to a loss of sodium bicarbonate and to an increase of sodium carbonate content. The reactive composition comprises generally at least 0.01 %, or at least 0.05 %, or at least 0.1 %, or at least 0.2 % by weight of water. The amount of water is measured by weight loss at a temperature of 25°C placing about 3g of the composition in a watch-glass and in
35 a dissicator in presence of silicagel under vacuum at about 50 mbar absolute pressure, during 24 hours. In present description 'free water' due to liquid water

absorbed on the composition (to be distinguished to linked water to the crystals of sodium carbonate such as for instance sodium carbonate monohydrate crystals) is measured by the loss of weight during 16 hours of a sample placed in a ventilated lab oven heated at 30°C.

5 The reactive composition according to the invention can also comprise one or more solid or liquid additives in order to improve, for example, the storage or flowability thereof, or to improve effectiveness of the product in flue gas treatment. Some additives can also further reduce the release of gaseous ammonia and ammonia smell by the composition at room temperature. The
10 additives are advantageously selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, active charcoal, fatty acids and fatty acid salts.

 The reactive composition according to the invention can preferably
15 comprise from 0.01 % to 5 % by weight of additives.

 Advantageously, the fatty acids are fatty acid molecules comprising 12 to 20 carbon atoms (C₁₂-C₂₀ fatty acid). More advantageously, the fatty acid is selected from lauric acid, myristic acid, palmitic acid, linoleic acid, oleic acid, stearic acid, and mixtures thereof. Stearic acid is preferred. It is particularly
20 advantageous that the corresponding fatty acid (i.e. the fatty acid or the fatty acid counter part of the fatty acid salt) have a melting point of less than 80°C, preferably less than 75°C.

 Fatty acid salts are advantageously selected from calcium, or magnesium acid salts or soaps of the fatty acids. More advantageously, the calcium or
25 magnesium fatty acid salts are selected from calcium or magnesium salt of : lauric acid, myristic acid, palmitic acid, linoleic acid, oleic acid, stearic acid, and mixtures thereof. Fatty acid salt is preferably selected from calcium stearate, magnesium stearate.

 The invention also relates to a process for the production of the reactive
30 composition according to the invention. It has been observed that a minimum temperature is needed to decrease the amount of water in compositions such as the ones described supra, in particular in crude sodium bicarbonate from an ammonia soda plant. In particular the invention relates to a process for the production of the composition according the present invention, according to
35 which particles resulting from crude bicarbonate particles from an ammonia-soda

plant are thermally treated at a temperature of more than 30°C. More particularly in this process :

- particles resulting from crude bicarbonate particles from an ammonia-soda plant are introduced into a gas stream generally comprising air in order to form a gas stream laden with particles;
- the gas stream laden with particles is introduced into a mill in order to form a gas stream comprising ground particles having a diameter D_{90} of less than 100 μm and a D_{50} of less than 75 μm , preferably a D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm measured by laser diffractometry.

In the present process, the temperature of the gas stream wherein the humid mixture comprising particles is introduced into, is preferably more than 40°C, more preferably more than 50°C, even more preferably more than 60°C, or more than 70°C, or more than 80°C.

In the present process the particles resulting from crude bicarbonate particles from an ammonia-soda plant comprises generally at most 20 % of water. It is advantageous that the crude bicarbonate particles comprise at most 15 %, preferably at most 12 %, more preferably at most 10 %, and even more preferably at most 8 %, or even at most 6 % in weight of water before being introduced into the gas stream. For lowering the water content of the particles resulting from crude bicarbonate particles, it is advantageous that the crude bicarbonate to be 'dewatered' in a dryer, such as a fluidized bed. In general the particles resulting from crude bicarbonate particles from an ammonia-soda plant comprises at least 2 %, more advantageously at least 3 % by weight of water when they are introduced into the gas stream.

In particular, such a minimum content of water (at least 2 %, more advantageously at least 3 % by weight of water) of the particles resulting from crude bicarbonate particles is beneficial when additives (selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, activated carbon or active charcoal, fatty acids and fatty acid salts), are added to the particles to form a humid mixture comprising particles and additives before being introduced in the gas stream or before the additives and the particles resulting from crude bicarbonate particles

are co-introduced into the mill. Indeed such water content of the mixture improves adhesion of the additives on particles of the composition and enables an improved adhesion of the additives on the particles resulting from crude bicarbonate particles.

5 In this process, the reactive composition is produced starting from crude bicarbonate particles from an ammonia-soda plant. This sodium bicarbonate is the product obtained by carbonation, with a gas comprising CO₂, of an ammoniacal brine. The particles formed at the end of the carbonation are separated from the slurry by filtration, in order to form the crude bicarbonate
10 particles from an ammonia-soda plant. The ammoniacal brine is obtained by reaction of ammonia with a sodium chloride solution. The crude bicarbonate from an ammonia-soda plant comprises predominantly sodium bicarbonate but also sodium carbonate, ammonia, some other compounds in small amounts and water. In the complete industrial process for the production of sodium
15 bicarbonate, the crude sodium bicarbonate is successively calcined (in order to produce "light" sodium carbonate, this calcination moreover producing CO₂), then dissolved, recarbonated with CO₂ and finally recrystallised. This transformation sequence exhibits a high cost, in particular a high energy cost (especially the calcination and recrystallisation). The use in the process
20 according to the invention of crude bicarbonate particles from a soda plant is thus of marked economic advantage. The inventors have observed that, when this product is ground in order to obtain a particle size distribution having a diameter D₉₀ of less than 100 µm and a diameter D₅₀ of less than 75 µm, preferably having a diameter D₉₀ of less than 50 µm and a diameter D₅₀ of less
25 than 35 µm, the amount of water can be decreased to values well below 1 % by weight without sensitively impacting the sodium bicarbonate content, which is not the case when drying coarse particles resulting from crude bicarbonate particles from an ammonia-soda plant. The ammonia content of the ground crude bicarbonate from a soda plant can be controlled with the temperature of the
30 thermal treatment and its duration. Moreover part of the gas stream from the thermal treatment remains generally at equilibrium around values of between 0.02 % and 0.2 % by weight, expressed as NH₄⁺.

 In some cases, for example when the ammonia content of the crude bicarbonate from a soda plant is too high, it is recommended for the crude
35 bicarbonate particles from an ammonia-soda plant to be washed using a washing liquid before being introduced into the gas stream.

In the present invention, it is generally recommended to remove the excess liquid, i.e. "dewatering" the crude sodium bicarbonate, for example by passing over a belt filter, in a centrifuge, a rotary filter or in a drying machine. The product can subsequently sometimes advantageously be dried. The drying of the particles can be realized on any appropriate equipment. Advantageously the drying is operated in a conveyor tunnel heated with hot gas, a fluid bed heated with hot gas, a fluidized bed heated indirectly with internal steam tubes, an agitated dryer, a rotary dryer, a direct heated rotary dryer with hot gas, an indirect heated rotary dryer heated with steam, a flash pneumatic conveyor dryer, or a gravity dryer. The drying can be performed as batch drying (loading the product in the dryer, drying and emptying the dryer) or as continuous drying operation (continuously feeding and continuously removing the dried product from the dryer). It is advantageous that the particles resulting from the crude bicarbonate after pre-drying comprises 2 % to 8 % of water, preferably 2 % to 6 % of water before said particles are introduced into the gas stream at a temperature of more than 30°C. For the drying operation the temperature is, for example, between 30 and 130°C or between 50 and 120°C, or preferably between 55 and 85°C. Stream gas up to 250°C can be used for the thermal treatment. Though the final temperature of the composition is generally lower due to water evaporation. The final temperature is preferably at most 130°C, preferably at most 100°C, more preferably at most 85°C, even more preferably at most 80°C. Then the dried product forming the composition generally comprises less than 1 % by weight of water, or at most 0.9 %, or at most 0.8 %, or at most 0.7 %, or preferably at most 0.5 %, more preferably at most 0.3 % by weight of water, and in particular by weight of free water. If high amount of sodium bicarbonate is desired, the contact time of the composition with hot gasses is to be limited. The more the temperature of hot gasses after water evaporation is, the shorter the contact time is to be. After the drying of the composition, it is recommended that the composition to be cooled down to at most 50°C, preferably to at most 40°C, more preferably to at most 35°C. This enables to avoid further bicarbonate decomposition and ammonia release when handling and storing the composition.

The gas stream comprising the ground particles resulting from the mill can be used directly, for example in the treatment of flue gases. However, in a recommended alternative form of the process according to the invention, the gas stream comprising the ground particles is introduced into a gas/solid separator in

order to provide, on the one hand, a gas stream comprising air and ammonia and, on the other hand, the reactive composition. The latter can then be supplied and used as is, for example in the treatment of flue gases, without requiring additional grinding or preparation. The reactive composition can be packaged, for example in bags, or stored in silos or containers.

It has been observed that the grinding operation in itself, probably as a result of the mechanical and thermal energy transferred to the reactive composition, has a favourable effect on its content of ammonia, which is then in equilibrium with the sodium bicarbonate and is further released only when the composition is heated. This effect is displayed in particular when the crude bicarbonate particles from an ammonia-soda plant have, before grinding, a diameter D_{90} of greater than 100 μm , preferably of greater than 120 μm , and a diameter D_{50} of greater than 60 μm , preferably of greater than 70 μm . In some cases, it can be advantageous to preheat the gas stream into which the particles are introduced, for example to a temperature of at least 45°C. As the grinding of the composition needs mechanical energy, part of this mechanical energy is dissipated during the milling into thermal energy rising the temperature inside the mill. A temperature of at least about 50°C in the mill is essential to decrease the amount of water to less than 1 % by weight of the thermally treated crude bicarbonate.

In an alternative form of the invention, the grinding is carried out in an impact mill. In the context of this alternative form, the impact mills are mills in which the material to be ground is subjected to the impact of moving mechanical parts which have the effect of smashing the particles of the material. Impact mills are well known in the art of fine grinding. They comprise in particular (nonexhaustive list) hammer mills or pin mills, "attritor" mills, bead mills and cage mills. Hammer mills are advantageously equipped with selectors.

As mentioned above, in order to improve the flowability and generally the flow properties of the reactive composition, one or more additives, advantageously selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, activated carbon or active charcoal, fatty acids and fatty acid salts, is (are) sometimes added to the composition. The fatty acid or fatty acid salt is preferably selected from calcium stearate, magnesium stearate and soaps. Some of these additives additionally have a beneficial effect during the use of the

reactive composition. For example, calcium carbonate, lime or activated carbon or active charcoals have a beneficial effect when the reactive composition is used in the treatment of flue gas, in particular for the purification from hydrogen fluoride.

5 In an advantageous alternative form of the process according to the invention, the solid or liquid additive or additives are introduced into the gas stream in an amount varying from 0.01 % to 20 % by weight. It is sometimes preferable for this introduction to take place upstream of the mill, or just when the gas stream enters the mill. This is because it has been observed that these
10 additives can also have a beneficial effect on the operation of the mill.

The invention also relates to the reactive composition which can be obtained by the process according to the invention. This composition is then obtained under highly advantageous conditions, starting from crude bicarbonate from an ammonia-soda plant. This composition is also obtained under very
15 favourable energy conditions, as it requires neither the calcination nor the recrystallisation which are necessary to produce the normal sodium bicarbonates. The energy saving has a positive environmental impact.

Finally, the invention also relates to a process for the purification of a flue gas comprising acidic impurities, for example hydrogen chloride or sulphur
20 oxides, according to which a reactive composition according to the invention, which can preferably be obtained by the process according to the invention, is introduced into the flue gas, at a temperature of 125 to 600°C, and the flue gas is subsequently subjected to a filtration. In particular flue gas comprising hydrogen chloride or sulphur dioxide are treated with the process according to the
25 invention so that hydrogen chloride be less than 10 or less than 5 mg HCl/Nm³ dry, and/or so that sulphur dioxide be less than 50 or less than 40 mg SO₂/Nm³ dry (at 11 % O₂). When the flue gas comprises nitrogen oxides ("NO_x"), the process advantageously comprises a catalyst in order to treat the NO_x, the catalyst being preferably incorporated into the filter. The ammonia content of
30 the reactive composition according to the invention has a beneficial effect on the operation of the catalyst, as in the hot flue gas the composition will release gaseous ammonia (NH₃), and by substitution up to 24 % of ammonia consumption can be saved.

Therefore the present invention also relates to a process for the purification
35 of a flue gas comprising acidic impurities, for example hydrogen chloride or sulphur oxides, according to which a reactive composition according the present

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invention is introduced into the flue gas, at a temperature of at least 100 or at least 125°C, and generally at most 600°C, the flue gas is subsequently subjected to a filtration, and then subsequently subjected to a selective catalytic reduction of nitrogen oxides (SCR DeNO_x).

5 In one embodiment of the process for the purification of a flue gas comprising dust, acidic impurities, such as hydrogen halides or sulphur oxides, and comprising nitrogen oxides (NO_x), the flue gas is optionally first dedusted so that to remove at least part of dust, then a reactive composition according present invention is injected in the at least partly dedusted flue gas so that to
10 absorb at least part of the acidic impurities, the resulting flue gas is then subsequently subjected to a filtration such as a bag filter to remove part of reacted reactive composition, and the flue gas is subsequently subjected to a selective catalytic reduction of nitrogen oxides (SCR DeNO_x).

In the purification process according to the invention, the filtration can be
15 performed using any filtering or separating means, for instance ceramic or metallic filters. Sleeve filters, in which the filtration takes place through a cloth, or electrostatic separators or multi cyclones are advantageous.

In the purification process according to the invention, it is recommended that the freeing of the flue gas from dust be carried out more than 2 seconds,
20 advantageously from 3 to 6 seconds, after the introduction of the reactive composition into the flue gas.

In the purification process according to the invention, the filtration equipment can integrate also the catalyst for the SCR DeNO_x operation. This, simplifies the process and reduces the investment costs.

25 **Example 1 (not according the invention)**

Particles of crude bicarbonate from an ammonia-soda plant, having a content of 74 % by weight of sodium bicarbonate, 9 % by weight of sodium carbonate, 0.8 % by weight of ammonia expressed as ammonium ions NH₄⁺, in the form of ammonium bicarbonate, ammonium carbonate, ammonium
30 carbamate, sodium carbamate, 15 % by weight of water, and having a particle size distribution such that the diameter D₅₀ has a value of 80 µm and the diameter D₉₀ has a value of 150 µm, are treated under air at 30°C.

The crude bicarbonate is heated in a glass reactor equipped with a jacket thermostatically controlled at 30 degrees centigrade.

After 2 hours, it is found that the value of the total ammonia no longer shows any substantial variation, even after treatment for approximately ten hours.

The final product, that is to say after treating for two hours, comprises
 5 84 % by weight of sodium bicarbonate, 10 % by weight of sodium carbonate, 0.6 % by weight of ammonia expressed as ammonium ions NH_4^+ , 3.4 % by weight of water, the remainder being composed of a negligible amount of sodium chloride.

Fifty grams of the product treated thermally is stored in a polyethylene
 10 bottle of 250 ml. After 24 hours of storage at room temperature (25°C) the polyethylene bottle is opened. The smell of ammonia is qualified as 'strong' by a laboratory operator, therefore with an ammonia concentration in the surrounding gas in equilibrium with the composition of more than 200 ppm NH_3 in volume.

15 **Example 2 (according to the invention)**

Particles of crude bicarbonate from an ammonia-soda plant, having a chemical composition similar to the one of example 1, having a particle size distribution such that the diameter D_{50} has a value of 80 μm and the diameter D_{90} has a value of 150 μm , are washed in a rotating filtering device fed continuously
 20 with water, constituting the washing liquid. An aqueous solution, comprising ammonia, is extracted from the rotating filtering device. The particles are subsequently introduced into a dryer. The particles, having a water content of less than 2 %, are introduced into a stream of air (7) itself entering a hammer mill with selector. The stream of air comprising the ground particles of sodium
 25 bicarbonate, gaseous ammonia and water vapour, both released from the particles during the grinding, is finally introduced into a sleeve filter. The following are extracted therefrom; on the one hand, a stream of air comprising water vapour and ammonia and, on the other hand, sodium bicarbonate particles having the properties shown in Table 1.

30

Table 1

D_{90}	D_{50}	NaHCO_3	Na_2CO_3	NH_4^+	H_2O
22 μm	9,6 μm	90 %	8 %	0.4 %	0.5 % (< 1 %)

The ammonia content of the sodium bicarbonate particles, expressed as NH_4^+ , has been evaluated by comparing their total NH_4 content before and after heating at 30°C during 2 hours. Total NH_4 was measured by alkaline distillation, described as being the second alternative determination.

Fifty grams of the product treated thermally according the process of present invention is stored in a polyethylene bottle of 250 ml. After 24 hours of storage at room temperature (25°C) the polyethylene bottle is opened. The smell of ammonia is qualified as 'light' by the same laboratory operator as example 1, therefore with an ammonia concentration in the surrounding gas in equilibrium with the composition of less than 150 ppm NH₃ in volume.

Example 3 (according the invention)

The example which is described below, with reference to the single appended figure, illustrates a specific embodiment of the invention.

Particles (1) of crude bicarbonate from an ammonia-soda plant, having a content of approximately 1 % by weight of ammonia and having a particle size distribution such that the diameter D₅₀ has a value of 80 µm and the diameter D₉₀ has a value of 150 µm, are washed in a rotating filtering device (A) fed continuously with water, constituting the washing liquid (2). An aqueous solution (3), comprising ammonia, is extracted from the rotating filtering device (A). The particles (4) are subsequently introduced into a dryer (B). The particles (5), having a water content of less than 2 %, are introduced into a stream of air (7) itself entering a hammer mill with selector (C). Amounts by weight (6) of two calcium based additives of totalling 1,2 % of the amount of particles (5) are also introduced into the mill (C). The stream of air (8) comprising the ground particles of sodium bicarbonate, gaseous ammonia and water vapour, both released from the particles during the grinding, is finally introduced into a sleeve filter (D). The following are extracted therefrom; on the one hand, a stream of air (9) comprising water vapour and ammonia and, on the other hand, sodium bicarbonate particles (10) having the properties shown in Table 2.

Table 2

D ₉₀	D ₅₀	H ₂ O	NaHCO ₃	Na ₂ CO ₃	Additives
22 µm	9,6 µm	< 1 %	> 85 %	< 11 %	1.1 %

The ammonia content of the sodium bicarbonate particles, expressed as NH₄⁺, has been evaluated by comparing their total NH₄ content before and after heating at 30°C during 2 hours. Total NH₄ was measured by alkaline distillation, described as being the second alternative determination.

The result was 0,3 g/kg.

The ammonia content of the particles exiting the rotating filtering device (A) was also measured. A value of 3,8 g/kg was found. Comparison with the value for the ground particles illustrates the effect of the invention.

Example 4 (according the invention)

5 120 kg of an ammoniacal sodium bicarbonate from Solvay process, exiting from a rotative filter after a carbonation column (such as described in Ullmann's Encyclopedia of Industrial Chemistry Sodium carbonate, page Vol. 33 page 307) comprising 13.7 % water is let to be dried on a polyethylene film one night at about 23°C to obtain a dewatered ammoniacal sodium bicarbonate with a water
10 content of about 6 % of water. The dewatered ammoniacal sodium bicarbonate is then introduced with hot air at 80°C into a pin mill UPZ100 Hozokawa Alpine. The obtained composition is then separated from the gas on a bag filter. The temperature of the gas exiting the bag filter is 55°C. The obtained composition comprises (weight percentage) : 89.8 % NaHCO_3 , 7.4 % Na_2CO_3 , 0.8 % total
15 ammonia expressed as NH_4^+ measured by the alkaline distillation method, 0.06 % water. The measured laser D_{90} is 25 μm .

Example 5 (according the invention)

200 grams of the similar samples of reactive compositions comprising different ammonia content and obtained as in example 3 (with less than 1 % of
20 water) are conditioned in a climatic chamber on a stainless steel plate with a powder thickness of 1.5 +/-0.5 cm, during 40 minutes, at different temperature and relative humidity (RH) conditions:

- 25°C and 40 % RH
- 25°C and 75 % RH
- 25 - 50°C and 75 % RH

Then part of the samples is introduced in 500 ml polyethylene bottles so that to fill about 250 ml of the reactive composition in powder form, and then to be able to measure the ammonia (NH_3) released in the atmosphere during storage.

30 The bottles are let during 40 minutes at room temperature (about 22°C) so that the atmosphere of the bottle is enriched in ammonia gas. Then the gas present in the polyethylene bottle is pumped out with a determined gas volume (corrected from measured ambient atmospheric pressure) through Draeger tube to measure the ammonia (NH_3) content of the gas within the storage bottle
35 (Draeger ammonia 5/a tubes range 5-600 ppm).

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Results obtained with the reactive compositions are reported in table 3
(25°C – 75 % RH).

Table 3 – Results with climatic conditioning at 25°C – 75 % RH
of Example 5

Samples	Nber	1	2	3	4	5	6
	Ref	3601-15	3628-14	3601-14	3624-15	3601-15	BNL
Total NH_4^+ content of sample %	% NH_4^+	0,48	0,61	0,58	0,38	0,64	0,96
NH_3 gas concentration	NH_3 ppm						
Crude Bicarb. (Example 4)	NH_3 ppm	150	200	200	100	150	> 610

Example 6

Similar tests as example 5, have been realized, showing the importance of low water content (less than 1% by weight) and particle size (D_{90} of less than 100 μm and D_{50} less than 75 μm) on the reactive composition to reduce ammonia release during storage and logistic handling of product with low particle size. A batch from crude sodium bicarbonate obtained from the ammonia Solvay process (comprising about 0.6% NH_4^+), was divided in three lots, each are partially dried at ambient temperature at respectively slightly above: 12%, 5% and less than 1% water content.

- 5 Each lot of reactive composition was conditioned 40 minutes at 25°C 75% relative humidity in a climatic chamber; for this, each lot was spread on a stainless steel plate with a layer of 1.5 +/- 0.5 cm of thickness.
- 10 Then part of the composition was taken and analyzed in remaining water content. 200 g sample of the pre-conditionned composition with measured and indicated water content in table 4, was introduced in polyethylene bottles of 500 ml volume, bottles were closed, put again in the climatic chamber at 25°C, and then after 40 minutes the bottles were opened and the gas in the bottles has been extracted with a pump and gas counter, and the gas was measured in ammonia content (expressed in ppm by weight). The result of ammonia concentration in the storage gas above the reactive composition is given in table 4.

- 15 Comparatively samples with at least 1% water content (5% and 11% water) of small particle size (D_{90} 50 μm , D_{50} 30 μm), showed an increased ammonia release during storage compared to small particle size with less than 1% water content. This was not the case for the same batch with coarser particles (before grinding): the ammonia release during the storage is the same for well
- 20 dried samples of coarse particle size (D_{90} 200 μm) whatever the water content (less than 1%; 0.2%, or 5% or 12% water) was. They release more ammonia gas in their storage environment, at ambient temperature (25°C) than samples of fine particle size and low water content.

30

Table 4 – Tests results of Example 6

Water content of samples	Coarse composition (D ₉₀ 200 µm)	Grinded composition with a mortar (D ₉₀ 50 µm, D ₅₀ 30 µm)
H ₂ O < 1 %	H ₂ O = 0.2 %wt NH ₃ odor : concentration in bottle gas = 50 ppm	H ₂ O = 0.2 %wt NH ₃ odor : concentration in bottle gas = 50 ppm
H ₂ O > 1%	H ₂ O = 12 %wt NH ₃ odor : concentration in bottle gas = 50 ppm H ₂ O = 5 %wt NH ₃ odor : concentration in bottle gas = 50 ppm	H ₂ O = 11 %wt NH ₃ odor : concentration in bottle gas = 75 ppm H ₂ O = 5 %wt NH ₃ odor : concentration in bottle gas = 75 ppm

CLAIMS

1. Reactive composition comprising between 60 % and 98 % by weight of sodium bicarbonate, between 1 % and 40 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , characterized in that the composition comprises less than 1.0 %, preferably less than 0.9 %, more preferably less than 0.8 % by weight of water measured by weight loss at a temperature of 25 °C placing about 3 g of the composition in a watch-glass and in a dissicator in the presence of silicagel under vacuum at about 50 mbar absolute pressure, during 24 hours.

2. Composition according to the preceding claim wherein the composition is in the form of particles having a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , measured by laser diffractometry.

3. Reactive composition comprising between 80 % and 98 % by weight of sodium bicarbonate, between 1 % and 20 % by weight of sodium carbonate and between 0.02 % and 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ , wherein the composition is in the form of particles having a diameter D_{90} of less than 100 μm and a diameter D_{50} of less than 75 μm , preferably a diameter D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm , measured by laser diffractometry, characterized in that the composition comprises less than 1.0 %, preferably less than 0.9 %, more preferably less than 0.8 % by weight of water measured by weight loss at a temperature of 25 °C placing about 3 g of the composition in a watch-glass and in a dissicator in the presence of silicagel under vacuum at about 50 mbar absolute pressure, during 24 hours.

4. Composition according to claims 1 or 2 comprising between 80 and 98 %, by weight of sodium bicarbonate.

5. Composition according to claims 1, 2 or 4 comprising between 1 % and 12 % by weight of sodium carbonate.

6. Composition according to one of the preceding claims comprising between 2 % and 12 % by weight of sodium carbonate.

7. Composition according to one of the preceding claims, comprising between 85 % and 95 % by weight of sodium bicarbonate.

8. Composition according to one of the preceding claims, comprising from 0.02 % to 0.17 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

9. Composition according to one of claims 1 to 7, comprising from 0.2 % to 0.7 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

10. Composition according to one of claims 1 to 7, comprising more than 0.7 % and less than 2.0 % by weight of ammonia, expressed in the form of ammonium ions NH_4^+ .

11. Composition according to one of the preceding claims, comprising from 0.01 % to 5 % by weight of additives selected from zeolites, dolomite, magnesium hydroxide, magnesium (hydroxy) carbonate, lime, calcium carbonate, sodium chloride, zinc chloride, sodium sulphate, calcium fluoride, hydrocarbons, talc, lignite coke, activated carbon or active charcoal, fatty acids and fatty acid salts.

12. Process for the production of a composition according to one of the preceding claims, according to which :

- particles resulting from crude bicarbonate particles from an ammonia-soda plant are introduced into a gas stream at a temperature of more than 30°C comprising air, in order to form a gas stream laden with particles;
- the gas stream laden with particles is introduced into a mill in order to form a gas stream comprising ground particles having a diameter D_{90} of less than 100 μm and a D_{50} of less than 75 μm , preferably a D_{90} of less than 50 μm and a diameter D_{50} of less than 35 μm , more preferably a diameter D_{90} of less than 35 μm and a diameter D_{50} of less than 20 μm , even more preferably a diameter D_{90} of less than 30 μm and a diameter D_{50} of less than 15 μm measured by laser diffractometry;
- to obtain a composition according to claims 1-11.

13. Process according to claim 12, in which the crude bicarbonate particles from an ammonia-soda plant have a water content of at most 15 % in weight, preferably at most 12 %, more preferably at most 10 %, and even more preferably at most 8 % in weight of water before being introduced into the gas stream.

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14. Process for the purification of a flue gas comprising acidic impurities, such as hydrogen chloride or sulphur oxides, according to which a reactive composition according to one of claims 1 to 11 is introduced into the flue gas, at a temperature of 100 to 600°C, and the flue gas is subsequently subjected to a
5 filtration or a dedusting.

15. Process according to claim 14 wherein the flue gas subjected to a filtration is, subsequently the filtration, subjected to a selective catalytic reduction of nitrogen oxides (SCR DeNO_x).