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(54)Title: PROCESS FOR PRODUCING CALCIUM CARBONATE (54)発明の名称 炭酸カルシウムの製造方法 (57) Abstract A process for producing calcium carbonate whose average major-axis length can be controlled. Wet slaking in which quick lime is slaked with water to prepare a slaked lime slurry is conducted in two steps. In the first step, water is reacted with quick lime in a changeable water/quick lime ratio in the range of 1.5 to 10 by weight. A slaked lime slurry is prepared through the first step, in which the water/quick lime weight ratio is regulated, and through the second step. Carbon dioxide is bubbled into the slurry to conduct carbonation. Thus, calcium carbonate particles with high evenness of size are produced in which the primary particles have an average major-axis length of 0.5 to 10 μm .		

SPECIFICATION

Process for Producing Calcium Carbonate

Technical Field to Which the Invention Pertains

The present invention relates to a process for producing calcium carbonate
5 comprising slaking quick lime with water to prepare slaked lime slurry, and reacting
the slaked lime slurry with carbon dioxide gas, by which the particle size of the
calcium carbonate can be controlled by controlling the reaction conditions.

Prior Art

The representative process for producing synthetic calcium carbonate is the
10 "liquid-gas" method in which calcium carbonate is produced by blowing carbon
dioxide gas into slaked lime slurry. It is known that calcium carbonate with various
particle sizes and various shapes can be produced by controlling the reaction
conditions such as concentration of the slaked lime slurry, reaction temperature,
method of reaction, presence or absence of additives, types of additives, if any, and
15 so on.

Problems Which the Invention Tries to Solve

However, a simple process for producing particulate calcium carbonate by
which the average longer diameter of the primary particles can be controlled within a
range of 0.5 to 2 μm , by the "liquid-gas" method that is widely employed in
20 industries, is not known. The particulate synthetic calcium carbonate of which
primary particles have an average longer diameter of 0.5 to 2 μm is suited as a
pigment or additive for coating compositions for papermaking, so that a production
process by which the particulate calcium carbonate can be produced inexpensively is
desired. To overcome this problem, a process in which seed crystals acting as
25 nuclei are added to the slaked lime slurry or to the partially carbonated slaked lime
slurry is described in Japanese Patent Publication (Kokoku) Nos. 7-5303 and 3-
19165. However, this process has a problem in that the overall process is

complicated because the seed crystals must be prepared in a separate step and the gas flow rate must be controlled.

5 A method in which partial carbonation is carried out in a condition under which pH is not lowered by not less than 0.2 to generate platelike calcium carbonate, and then carbonation is continued until the reaction is completed, is described in Japanese Patent Publication (Kokoku) No. 3-3605. However, with this method, the flow rate of carbon dioxide gas should be restricted and should be changed depending on the pH value, so that the method is time-consuming and complicated.

10 A method in which partially carbonated slaked lime slurry is ground under wet condition and then secondary slaked lime slurry is added to complete carbonation is described in Japanese Laid-open Patent Application (Kokai) No. 5-221634. However, since the grinding under wet condition is carried out using a sand grinder after partial carbonation, the overall process is complicated.

15 On the other hand, "liquid-liquid" process is a process in which a solution containing carbonate ion (aqueous sodium carbonate solution, aqueous ammonium carbonate solution or the like) and a solution of a calcium compound (aqueous calcium chloride solution, aqueous calcium acetate solution or the like) are mainly reacted to obtain calcium carbonate particles. A process for obtaining particulate calcium carbonate having a particle size of 0.1 to 20 μm is described in Japanese
20 Laid-open Patent Application (Kokai) No. 7-196316, in which a carbonate solution containing a reaction buffer and a calcium salt solution are mixed by dropping one of these solutions to the other so as to carry out carbonation reaction, and an aqueous salt or an aqueous acid which forms a water-insoluble salt or relatively water-insoluble salt upon reaction with calcium is dropped to the mixture. However, to
25 carry out this process, a material tank is needed for each material and the rate of dropping must be controlled, so that the overall equipment for carrying out this process is complicated.

Problems Which the Invention Tries to Solve

Accordingly, an object of the present invention is to provide a process for producing calcium carbonate by which a particulate calcium carbonate whose particle size is controlled to a desired size can be obtained simply and inexpensively.

Summary of the Invention

According to a first embodiment of the invention there is provided a process for producing calcium carbonate comprising

a two-step slaking step including a first step of reacting water and quicklime, in which the water ratio may be changed, and a second step of preparing a slaked lime slurry having a prescribed concentration and aging the resultant; and

a carbonation step in which carbon dioxide is blown into said slaked lime slurry prepared in said slaking step, wherein

the water/quick lime weight ratio in the first step of said slaking step is 1.5 to 10.

The invention also provides calcium carbonate produced according to the process of the first embodiment of the invention.

Brief Description of the Drawings

Fig. 1 is an electron microphotograph of calcium carbonate obtained in Example 2.

Fig. 2 is an electron microphotograph of calcium carbonate obtained in Example 4.

Fig. 3 is an electron microphotograph of calcium carbonate obtained in Comparative Example 1.

Fig. 4 is an electron microphotograph of calcium carbonate obtained in Comparative Example 2.

Modes for Carrying Out the Invention

The present invention is based on the discovery made by the present inventors that by changing the water ratio within the range of 1.5 to 10 in the first step of the slaking step, the particle size of the produced calcium carbonate is changed depending on the water ratio. More particularly, it was proved that if the water ratio is within the range of 1.5 to 10, particles having an average longer diameter of the

primary particles of 0.5 to 2 μm , which longer diameter is measured by the method described below, which particles have a substantially uniform particle size, are obtained.

In the present invention, the water ratio in the first step of the slaking process is controlled within the range of 1.5 to 10 depending on the desired particle size of calcium carbonate. Within the range of 1.5 to 10, the larger the water ratio, the larger the particle size of the calcium carbonate obtained. If the water ratio is less than 1.5, vaporous slaking occurs during the slaking, so that the particle size of the obtained slaked lime is extremely large and the calcium carbonate produced in the later step is coagulated. On the other hand, if the water ratio is more than 10, the produced calcium carbonate has a poor dispersibility, which is not desired from the viewpoint of quality of the product.

In a preferred mode, to obtain calcium carbonate particles with high uniformity, in the first step of the slaking process, from substantially the beginning of the slaking reaction, and in substantially the entire region in which the slaking reaction occurs, the prescribed amounts of quick lime and water are contacted such that the slaking is carried out under a specific water ratio selected from the range of 1.5 to 10. That is, simultaneously or immediately after commencement of the reaction, the reaction is carried out under a substantially specific water ratio. More particularly, for example, a prescribed amount of quick lime is added to a prescribed amount of water within about one minute from the beginning of the addition. If the time period of adding quick lime to the prescribed amount of water is more than one minute, the raise of the initial slaking temperature is slow and so the slaking reaction is also slow, so that the slaked lime particles are likely to coagulate. This causes a problem that the calcium carbonate particles generated from the slaked lime are also likely to coagulate, which is not desired from the viewpoint of quality of product for some uses of the calcium carbonate. In case of industrial production, it may be

necessary for controlling the rate of adding quick lime, so that an appropriate rate may be selected depending on the amount of the lot, reaction conditions such as method of slaking and temperature, and on the uniformity of the particles and the allowable degree of coagulation, demanded for a commercial product.

5 The two-step wet slaking process comprises a first step of reacting water and quick lime in which the water ratio is selected, and a second step of adjusting the slaked lime slurry to a prescribed concentration and aging the resultant. Either continuous two-step wet slaking or batch type two-step wet slaking can be employed in the present invention.

10 The slaking in the first step in the two-step wet slaking process may be carried out by batch type slaking bath method, continuous mixing method using paddles, continuous mixing method using screws, continuous mixing method using kneader or the like. As long as the condition in which the slaking is carried out under a specific water ratio is attained, any slaking method may be employed. To add a
15 prescribed amount of quick lime substantially simultaneously, addition in one batch from a hopper with a meter may be employed.

 Since the second step of the slaking process is for adjusting the slurry to a prescribed concentration and for aging the slurry, the method is not restricted.

 Before the carbonation reaction in the carbonation step, the concentration of
20 the slaked lime slurry is preferably adjusted to 3 to 15% by weight and the temperature thereof is preferably adjusted to 30 to 90°C. Carbon dioxide gas or a mixed gas containing carbon dioxide gas is blown into this calcium hydroxide slurry until the ratio of carbonation reaches substantially 100%. Similar to the conventional production process of calcium hydroxide, if the concentration of
25 calcium hydroxide is less than 3% by weight, the industrial productivity is poor, and if the concentration is more than 15% by weight, the viscosity of the slurry is so high that ease of handling is poor. If the temperature of the slaked lime slurry before the

reaction is lower than 30°C, colloidal calcite is likely to be generated to coagulate the particles, and that if the temperature is higher than 90°C, energy cost is high, which is disadvantageous. The carbonation step may be either continuous process or batch process.

5

Effects of the Invention

By the process for producing calcium carbonate according to the present invention, a particulate calcium carbonate of which primary particles have a controlled average longer diameter within the range of 0.5 to 2 μm can be obtained. The process for producing calcium carbonate according to the present invention can be carried out using the conventional equipment substantially without modifying the conventional equipment or without adding a new apparatus thereto, the process can be realized simply and inexpensively without a high cost equipment investment.

10

15

The calcium carbonate produced by the process according to the present invention has an excellent uniformity in the particle size. Therefore, when the calcium carbonate is used as a pigment in the coating composition or an inner filler used for papermaking, various properties such as whiteness, luster and smoothness are expected to be improved.

Examples

20

The present invention will now be described by way of examples thereof. Needless to say, the present invention is not restricted to the following examples and various modifications can be made within the scope of the gist of the present invention.

Example 1

25

Quick lime blocks (JIS industrial lime, special grade quick lime, particle size 3 to 35 mm) produced by the applicant company were used as the material quick lime. In the first step in the slaking process, the quick lime was added to water at 70°C such that the water/quick lime ratio was 1.5, and the resulting mixture was allowed to

react. In the second step in the slaking process, concentration and temperature were adjusted and aging was carried out to obtain a slaked lime slurry having a temperature of 60°C and a concentration of 5% by weight. Into this slurry, carbon dioxide-containing gas (CO₂ concentration: 20 vol%) was introduced at a flow rate of 34L/min · kg-Ca(OH)₂ until the ratio of carbonation reaches 100%, thereby completing the reaction to obtain calcium carbonate.

The obtained calcium carbonate was observed with an electron microscope. As a result, it was confirmed that the calcium carbonate was in the form of uniform particles having an average longer diameter of the primary particles of 0.5 μm.

The term "average longer diameter of primary particles" means the average of the length of the longest chord of the particles on the two diagonal lines of the electron microphotograph randomly taken at a magnification of 30,000x, which length of the longest chord was measured with an electron caliper.

Example 2

Calcium carbonate was produced in the same manner as in Example 1 except that the water/quick lime ratio in the first step of the slaking process was 3. By observation with an electron microscope, it was confirmed that the calcium carbonate was in the form of uniform particles having an average longer diameter of the primary particles of 1 μm.

Example 3

Calcium carbonate was produced in the same manner as in Example 1 except that the water/quick lime ratio in the first step of the slaking process was 5. By observation with an electron microscope, it was confirmed that the calcium carbonate was in the form of uniform particles having an average longer diameter of the primary particles of 1.3 μm.

Example 4

Calcium carbonate was produced in the same manner as in Example 1 except

that the water/quick lime ratio in the first step of the slaking process was 7. By observation with an electron microscope, it was confirmed that the calcium carbonate was in the form of uniform particles having an average longer diameter of the primary particles of 1.7 μm .

5 Example 5

Calcium carbonate was produced in the same manner as in Example 1 except that the water/quick lime ratio in the first step of the slaking process was 10. By observation with an electron microscope, it was confirmed that the calcium carbonate was in the form of uniform particles having an average longer diameter of the primary particles of 2 μm .

10 Comparative Example 1

Calcium carbonate was produced in the same manner as in Example 1 except that the water/quick lime ratio in the first step of the slaking process was 1. By observation with an electron microscope, it was confirmed that the calcium carbonate was in the form of non-uniform coagulated particles having an average longer diameter of the primary particles of 0.5 μm .

15 Comparative Example 2

Calcium carbonate was produced in the same manner as in Example 1 except that the water ratio in the slaking process was 26, and the slaking process was carried out in one continuous step, which is usually employed. By observation with an electron microscope, it was confirmed that the calcium carbonate was in the form of non-uniform coagulated particles having an average longer diameter of the primary particles of 2.5 μm .

20
25 Table 1 summarizes the relationship between the water ratio and the particle size of calcium carbonate in each Example and Comparative Example.

Table 1

	Water/Quick Lime Weight Ratio [-]	Median Radius Measured by Laser Diffraction Method* [μm]	Average Longer Diameter of Primary Particles [μm]
Comparative Example 1	1	6.4	0.5
Example 1	1.5	2.5	0.5
Example 2	3	2.9	1.0
Example 3	5	3.1	1.3
Example 4	7	3.4	1.7
Example 5	10	4.1	2.0
Comparative Example 5	26	7.6	2.5

*: Apparatus used for measurement: HORIBA LA-500

The claims defining the invention are as follows:

1. A process for producing calcium carbonate comprising
a two-step slaking step including a first step of reacting water and quicklime, in
which the water ratio may be changed, and a second step of preparing a slaked lime slurry
5 having a prescribed concentration and aging the resultant; and
a carbonation step in which carbon dioxide is blown into said slaked lime slurry
prepared in said slaking step, wherein
the water/quick lime weight ratio in the first step of said slaking step is 1.5 to 10.
2. The process according to claim 1, wherein said two-step wet slaking process
10 is a continuous process.
3. The process according to claim 1, wherein said two-step wet slaking process
is a batch process.
4. The process according to any one of claims 1 to 3, wherein said calcium
carbonate produced has an average longer diameter of primary particles of 0.5 to 2 μ m.
- 15 5. The process according to any one of claims 1 to 4, wherein slaking in the first
step of said slaking step is carried out under the water/quick lime weight ratio of 1.5 to 10
substantially from the beginning of the slaking reaction, said water/quick lime weight
ratio being attained in substantially the entire region in which the slaking reaction occurs.
6. A process for producing calcium carbonate, substantially as hereinbefore
20 described with reference to any one of the examples but excluding the comparative
examples.
7. Calcium carbonate produced according to the process of any one of claims 1
to 6.

Dated 16 October, 2003
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FIG. 1

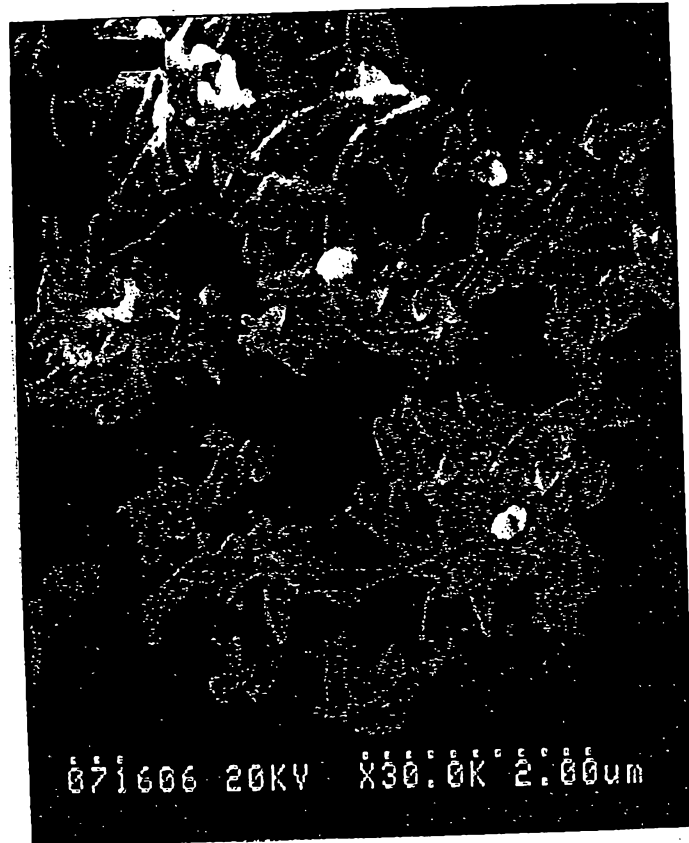


FIG. 2



FIG. 3

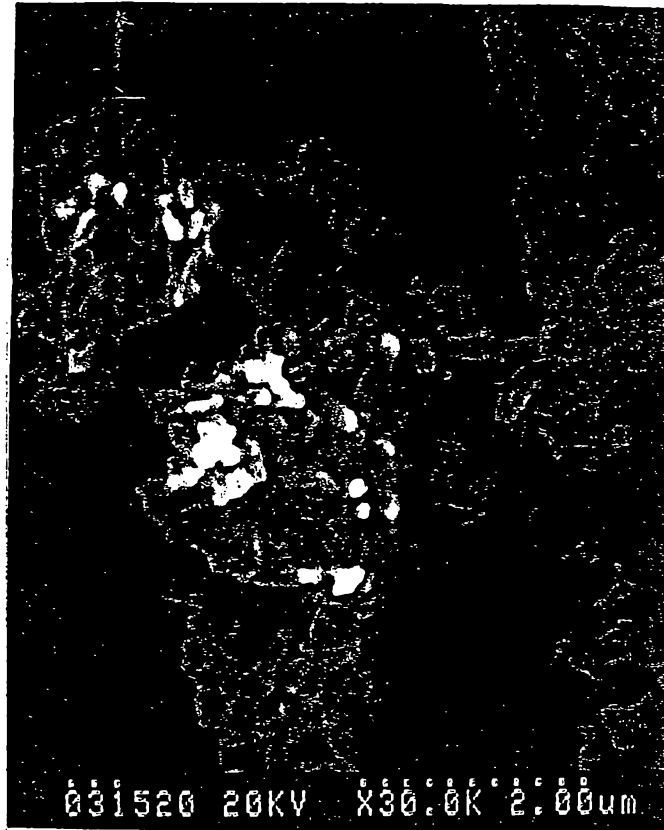


FIG. 4

