Title: METHOD FOR PULVERIZING NATURAL ORGANIC SUBSTANCE INTO NANO-SCALE FIBROUS MATERIAL

Abstract: A method for pulverizing natural organic substance into nano-scale fibrous material including steps: Cleaning raw material of natural organic substance from grease and drying it with common equipments. Coarsely pulverizing said prepared natural organic substance with common mechanical milling machine into small piece of a particle size around 500 μm in length. Mixing said coarse particles with pure water as a suspension and finely pulverizing it by a professional ultrasonic crusher into fine piece of a particle size smaller than 20 μm both in diameter and length. Feeding said fine particles contained suspension with high pressure into a nano collide machine for dividing said suspension into two parts to collide with each other and generate vibration to cause said raw material into nano-scale size. Drying said nano particles with a common spray dryer if dry nano-scale organic substance fibrous particle is in need.
For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.
Method for Pulverizing Natural Organic Substance into Nano-scale Fibrous Material

Field of the invention

This invention relates to pulverization process, especially to pulverizing natural organic substance into nano-scale fibrous material.

Background of the invention

Natural fibers have played an important role in textile material from ancient time and still have been widely used in the modern textiles industry for their unique properties as high quality textile material. However, for the limitation/requirement in the stage of spinning, not all fibers can be used to spin, because of their short length. Therefore, sometimes there are some natural fibers such like wool, silk, cotton or hemp etc. is wasted during processing. To develop a new way to reuse those fibers has large marketing potential because of their excellent intrinsic properties. Meanwhile, not only textile industry, many other industries also need such technology in new material or functional design or application.

There were many prior arts tried to reuse said short length fibers, such as US 6,437,050 B1 disclosed a polymer nano-particle composition with a poly (alkenybenzene) core and a surface layer including poly(conjugated diene) by a dispersion polymerization process with the average size less than 100 nm in diameter. CN 94115873.X disclosed a method to prepare nano-scale cellulous powder with an average size of 2.5 -- 10 nm from cotton or hemp fibers by chemical treatment together with low temperature drying and pulverization and filtration. These US and CN prior arts taught us specific methods to prepare few specific nano organic powders. The method of how to produce nano-scale particles from various natural organic substances has not yet published.

Usually organic material exhibit high strength and high elongation thus resulting in high destroy energy, they are difficult to be crushed/grinded/pulverized by traditional steel or iron milling machine into fine particle size. Mean while, traditional metal machine will generate lots of energy during the milling, and also are very easy to be worn out compared with organic material if under long time milling.
Summary of the invention

The object of this invention is to provide a method for pulverizing natural organic substance into nano-scale fibrous materials.

For achieving this object, this invention provides a new method for pulverizing the organic substance into nano-scale fibrous materials with combination of several different crushing technologies to meet the different requirements at each stage. With this method, the organic substance can easily and effectually to be crushed into nano-scale super fine fibrous particles. The method includes the following steps:

1. Raw material preparation: natural organic substance is cleaned and dried by common equipments. For example, a commercial wash machine and a dryer can be used.

2. First pulverization: The organic substance is crushed into small piece by a mechanical milling machine (for example, a rotate crusher). After this process the particle size around 500 μm in length can be achieved.

3. Second pulverization: The product from step 2 is mixed with pure water as a suspension and pulverized by a professional ultrasonic crusher. After this pulverization, the size of the organic particle in the suspension will be smaller than 20 μm both in diameter and length.

4. Third pulverization: The suspension containing the particles from step 3 is fed into a nano collide machine for dividing said suspension into two parts to collide with each other and generate vibration to cause said natural organic substances into nano-scale size. After this step, the size of the particles will be smaller than 100 nm in diameter and less than 800nm in length. The particle size can be controlled by adjustment of the collide pressure and collide times.

The following step will be further required, if dry nano-scale organic substance fibrous particles are in need. The step is:

5. Dry fibrous particle preparation: drying the nano particles from step 4 with a common spray dryer to produce dry nano-scale organic substance fibrous particles.

Brief description of drawings

Fig. 1a is a close view of a wool fiber, about 25 μm in diameter and 60 -120 mm in
length.

Fig. 1b is a close view of said wool fiber after first pulverization, the size is now reduced to 0.5 mm in length.

Fig. 1c is a close view of said wool fiber after second pulverization, the size is now reduced to 20 μm in length and in diameter.

Fig. 1d is a close view of said wool fiber after third pulverization, the size is now reduced to 50 nm in diameter and 200 -- 800 nm in length.

Fig. 1e is 3D SPM image for wool fibrous particle.

Fig. 2a is continuous strand of silk protein filament and 15-30μm in diameter.

Fig. 2b is a close view of said silk fiber after first pulverization, the size is now reduced to 0.5mm in length.

Fig. 2c is a close view of said silk fiber after second pulverization, the size is now reduced to less than 20 μm in length and in diameter.

Fig. 2d is a close view of nano scale silk particles after third pulverization.

Fig. 3a is a close view of cotton fiber after first pulverization, the size is about 0.5mm in length.

Fig. 3b is a close view of said cotton fiber after second pulverization, the size is reduced to less than 20 μm in length and in diameter.

Fig. 3c is a close view of nano scale cotton particles after third pulverization.

Fig. 4a is a close view of scutellaria particle after first pulverization, the size is about 0.5mm in length.

Fig. 4b is a close view of said scutellaria particle after second pulverization, the size is reduced to less than 20 μm in length and in diameter.

Fig. 4c is a close view of nano scale scutellaria particles after third pulverization.

Fig.4d shows the SEM image for nano scale scutellaria particles deposited on a cotton fiber surface.

**Detailed Description of the Preferred Embodiments**

As an example, wool fiber, which has about 60-120 mm in length and around 25 μm in diameter, is used to be pulverized following the method described above and the microscope
images at different steps are shown from Fig.1a to Fig. 1e.

Fig. 1a shows wool fiber, about 60-120 mm in length and 25 μm in diameter. For performing step 1, wool fibers must be enwrapped with mesh bags. Any detergent such as sodium dodecyl sulphate or anionic surface-active agent can be used for taking away the grease from the wool fiber. In this embodiment, first to add detergent (like soap or dishwashing etc.) in the washer and put warm water (50-90°C preferred) in a large tub or washer to submerge the wool, but don’t agitate it. Let the wool sit in the soapy water for about a half hour. Then set the washer on the spin only cycle and put the wool in, then adding the clean water and spin again. Finally, take the wool out and dry it with a common equipment.

Fig. 1b shows the particle at size of about 0.5 mm in length after first pulverization. In step 2, said first pulverization is a coarse pulverization. A range of mechanical crushers can be used to crush the organic materials from step 1 into small size particles as mentioned. For example, FZ102, a lab mini rotary crusher can be used to pulverize the wool fiber into small segments.

Fig. 1c shows most wool fibrous particle is crushed into smaller particle and the size is less than 20 μm in length and in diameter after second pulverization (600W, 270 Second). In step 3, for further pulverization, the coarse particle from step 2 is mixed with pure water as a suspension and pulverized by a professional ultrasonic crusher. A range of professional ultrasonic crushers can be used in this step, for example, an ultrasonic crusher JY92-II. It was set at a cycle of running for 3 seconds and stopping for 2 seconds under the output crush power at 600W. In this embodiment, total 270 seconds were used. But the operation parameters should be set according to the kind of organic materials to be crushed.

Fig. 1d shows wool particle at size of smaller than 50 nm in diameter and 200 to 800 nm in length after being crushed one time by a collide machine under a pressure of 1000kgf /cm². In step 4, for performing the final pulverization, the principle of high pressure coupled with vibrations is used. For instance, a NT1500/5 Nano collide machine designed with such principle is used to pulverize wool particles into nano scale fibrous materials. Raw materials pressurized by the high-pressure pump and fed into Nano collide machine through the high-pressure tubing. In the Nano collide machine, the raw materials are divided into two
parts to collide with each other and generate vibration to complete the pulverization in the moment. For example, the machine was set at 1000kgf/cm² pressure working condition and wool fiber particles can be crushed to the size less than 100 nm in diameter. The particle size can be controlled by adjustment of the collide pressure and collide times.

Fig. 1e shows 3D SPM image for wool fibrous particle. In step 5, spray dryer is an optional item, which is needed only when the dry particles are required. A range of spray drying machines can be used. For example, a Mini Spray Dryer can be used with setting of 2900W, temperature 100°C Compressed air 5 bar.

Wool fibrous particles presented in the invention have been produced and the particle size also tested. The test results can be found in the corresponding images.

In addition, the silk particles, cotton particles and scutellaria particles with nano-scale size are obtained by the method for pulverization of natural organic substance into nano-scale fibrous materials of this invention, and the microscope images of the particles in different steps are shown respectively from Fig.2a to Fig. 2d for silk, from Fig.3a to Fig. 3c for cotton, and from Fig.4a to Fig. 4d for scutellaria.

The method presented in this invention is a new way to pulverize the organic substance into nano-scale particle with combination of several different crushing technologies to meet the different requirements at each stage. With this method, we can easily and effectually to crush the organic substance into nano-scale super fine fibrous particle.

It is to be understood that the foregoing described embodiments of wool, silk, cotton and scutellaria are not intended to limit the scope of the invention. The method of the present invention can be used for any natural organic materials to be pulverized into nano-scale size.

Specification of Equipments used in this embodiment for natural organic substance pulverization

1. FZ102 lab mini rotary crusher
   a. Voltage 220 V
   b. Motor Power 150 W
   c. Rotational speed 1400 rpm
   d. Mesh 0.5 mm

2. JY92-II ultrasonic crusher
   a. Supply Voltage 220 V
   b. Power 650 W
c. Capacity 0.5-500 ml
d. Duty Ratio 0.1-99%

3. Collide machine
   a. Supply voltage 380 V
   b. Power 1.5 KW
   c. Max Pressure 1500 Mpa
   d. Max Capacity 5 L/hr

4. Spray Dryer
   a. Supply Voltage 380 V
   b. Powder 2900 W
   c. Compressed air 5 Bar
Claims

1. A method for pulverizing natural organic substance into nano-scale fibrous material, wherein said method includes the following steps:
   1. Raw material preparation: cleaning natural organic substance from grease and drying with common equipments;
   2. First pulverization: coarsely pulverizing prepared natural organic substance from step 1 with common mechanical milling machine into small piece of a particle size around 500 µm in length;
   3. Second pulverization: mixing coarse particles from step 2 with pure water as a suspension and finely pulverizing it by a professional ultrasonic crusher into fine piece of a particle size smaller than 20 µm both in diameter and length;
   4. Third pulverization: feeding fine particles contained in the suspension from step 3 at high pressure into a nano collide machine for dividing said suspension into two parts to collide with each other and generate vibration to cause said natural organic substances into nano-scale size.

2. A method for pulverizing natural organic substance into nano-scale fibrous material as per claim 1, wherein said method further includes drying nano particles from step 4 with a common spray dryer to obtain dry nano scale organic substance fibrous material.

3. A method for pulverizing natural organic substance into nano-scale fibrous material as per claim 1, wherein said common equipments include any commercial wash machine, a dryer, a lot of mesh bags and any detergent which can take away the grease.

4. A method for pulverizing natural organic substance into nano-scale fibrous material as per claim 2, wherein said detergent includes soap or dishwashing.

5. A method for pulverizing natural organic substance into nano-scale fibrous material as per claim 1, wherein said common mechanical milling machine includes rotary crusher.

6. A method for pulverizing natural organic substance into nano-scale fibrous material as per claim 1, wherein said professional ultrasonic crusher includes JY92-II.
## INTERNATIONAL SEARCH REPORT

### A. CLASSIFICATION OF SUBJECT MATTER

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According to International Patent Classification (IPC) or to both national classification and IPC.

### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

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<th>IPC</th>
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Documented searched other than minimum documentation to the extent that such documents are included in the fields searched.

China Inventions, China Utility Model

Electronic database consulted during the international search (name of database and, where practicable, search terms used)

WPI, EPDOC, PAJ

pulv+; organic, substance, wool, nano, fiber, fibrous, mix+, suspension

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
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<td>A</td>
<td>CN1104647A 5.Jul.1995 (5.7.1995) See the whole document</td>
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<td>PA</td>
<td>CN1416745A 14.May.2003(14.5.2003) See the whole document</td>
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☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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  - **A** document defining the general state of the art which is not considered to be of particular relevance
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