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(71) Applicant: Nippon Gohsei Kagaku Kogyo Kabushiki Kaisha
No 9-6, Nozaki-cho, Kita-ku
Osaka-shi, Osaka-fu(JP)

(72) Inventor: Fujita, Shigemi
1577-11, Tsuya Nanno-cho
Kaizu-gun Gifu-ken(JP)

(72) Inventor: Ozaki, Nobuyuki
523-5, Kasago-cho
Ogaki-shi Gifu-ken(JP)

(74) Representative: Gille, Christian, Dipl.-Ing. et al,
Türk, Gille + Hrabal Patentanwälte Bruckner Strasse 20
D-4000 Düsseldorf 13(DE)

(54) Treating apparatus having multifunction.

(57) A treating apparatus having multifunction comprising a vessel having inverted frustoconical configuration with a vertical central axis; a stirring assembly with spiral blade disposed in the vessel adjacent to an internal peripheral surface thereof; and a driving and supporting assembly which rotates the stirring assembly around its own axis and revolves the stirring assembly around the central axis wherein a filter medium comprising porous material is disposed at the bottom of the vessel.

According to an apparatus of the present invention, there can be remarkably improved purification process comprising an operation of drying powdery or granular substance, an operation of drying, and the like.

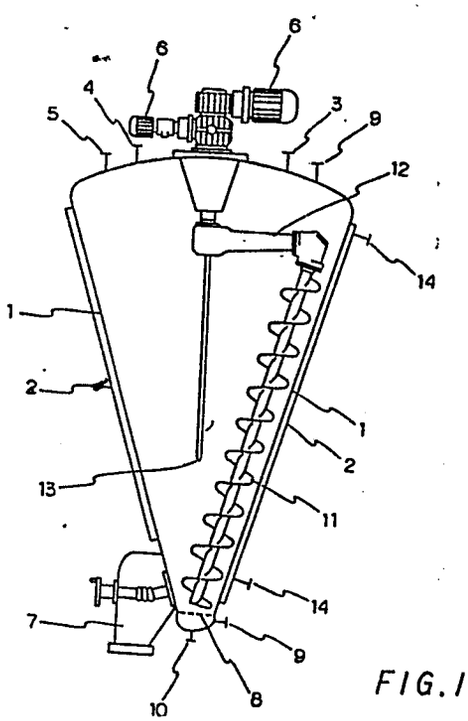

FIG. 1

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TREATING APPARATUS HAVING MULTIFUNCTION

The present invention relates to a treating apparatus having multifunction adaptable for multiple-purpose treatment of powdery or granular substance, and more particularly to a treating apparatus having multifunction wherein there can be carried out various operations such as mixing, washing, extraction, filtration and drying of powdery or granular substance in a single vessel which also functions as a hopper.

There has been widely employed a process comprising unit operations such as mixing, washing, extraction, filtration, drying and packaging of powdery or granular substance in chemical industry, medicinal industry, food industry, and the like. The process has important influence upon the quality or commercial value of products.

Hetherto, in a process from purification of powdery or granular reaction product to packaging thereof in, for example, chemical industry, there have been carried out successive unit operations such as crystallization out of solution, separation, washing, separation, drying, storage in hopper and packaging by using individual apparatus. In that case, there were required a great deal of hands or transport equipments each of which has specialized function corresponding to properties of powdery or granular substance in order to transport powdery or granular slurry (dispersion) or wet cakes (wet or coagulative powder or granular substance) between each apparatus.

In case that the above-mentioned trasportation between each apparatus is carried out automatically, there is generated contamination (such a contamination

that is caused by mixing of undersirable material) of products whenever an object to be treated changes and it is necessary, therefore, to wash the whole system to prevent the contamination.

Recently in particular there is a tendency that fine chemical products are widely required and many kinds of chemical products are required in a small lot, and accordingly there is required to wash the whole system at the cost of operating time or to newly set up an treatment process exclusive to each product or each grade of product in order to secure the purity of products, which is a main factor to increase production cost.

In solving the above-mentioned problems, we inventors considered the necessity of lessening transportation times between each apparatus as much as possible in the purification process of powdery or granular substance, and so vigorously studied a treatment apparatus having multifunction thereby there can be carried out in a single vessel such treatment operations that have been performed by using individual treatment appratus as to complete the apparatus of the present invention.

Accordingly, it is an object of the present invention to provide a treating apparatus having multifunction wherein there can be carried out various operations such as mixing, washing, extraction, filtration and drying of powdery or granular substance in a single vessel which also functions as a hopper.

It is another object of the present invention to provide improved processes for purification of reaction products such as intermediates for medicines which are strictly required not to contain foreign matter, purification of materials which are easily deteriorated by contact with air or by exposure to light, purification operation in which toxic, poisonous or hazardous solvents are employed, purification operation which contains crystallization by concentration, heterogeneous reaction of high polymers such as acetoacetalization of

polyvinyl alcohol (cf. Japan Patent No. 1,196,349) and purification of reaction products of powdery or granular high polymers.

In accordance with the present invention, there is provided a treating apparatus having multifunction comprising a vessel having inverted frustoconical configuration with a vertical central axis; a stirring assembly with spiral blade disposed in the vessel adjascent to a internal peripheral surface thereof; and a driving and supporting assembly which gives stirring assembly a planetary motion, wherein a filter medium comprising porous material is disposed at the bottom of the vessel. In an apparatus of the present invention, many kinds of functions such as washing, extraction, filtration and drying are added to a stirring apparatus besides a function of mixing of powdery or granular substance.

The above and other objects of the present invention will be seen by reference to the description taken in connection with the accompanying drawing.

Fig. 1 is a view explaining a fundamental notion of an apparatus of the present invention.

Referring now to an accompanying drawing, features of an apparatus of the present invention is explained in detail.

In an apparatus of the present invention, a jacket 2 for heating or cooling is disposed at outer surface of a sidewall of a vessel 1 having inverted frustoconical configuration with a vertical central axis. There is provided a pair of ports 14 for steam or warm water used for heating, or for cold water used for cooling on the jacket 2.

At the top portion of the vessel 1, there are disposed a inlet port 3 for powdery or granular substance or slurry thereof, a charge port 4 for liquid to wash or extract, a discharge port 5 for vapour which is generated during the drying of wet cake or for drying gas, and a motor assembly 6 for driving the stirring assembly.

At the lower portion of the vessel 1, there is disposed an outlet port 7 for powdery or granular substance treated.

A filter medium 8 comprising porous material is disposed at the bottom of the vessel 1, and below the filter medium 8 there are provided an inlet port 9 for drying gas and a discharge port 10 for filtrate. Inlet ports 9 can also be disposed at the top portion of the vessel 1 if desired.

A stirring assembly 11 with spiral blade is disposed adjascent to the internal peripheral surface of the vessel 1. The top portion of the stirring assembly 11 is rotatably installed at the outer end portion of a supporting arm 12 which extends outwardly in radial direction. The stirring assembly 11 is driven by a motor assembly 6 for driving. The lower portion of the stirring assembly is usually unsupported as shown in Fig. 1, it might be, however, supported by a bearing means mounted at the bottom of the vessel 1 if desired.

There is provided a temperature sensor 13 at a revolving shaft of the supporting arm 12.

In an apparatus of the present invention, it is necessary to employ a vessel 1 having inverted frustoconical configuration with a vertical central axis in order to improve the efficiency of mixing and heat transfer. The vertical angle of the inverted frustoconical configuration is preferably 30 to 40 degrees. The shape of a filter medium 8 in the apparatus of the present invention is optionally selected from the disc, conical, hemispheric, and the like depending on the properties of powdery or granular substance to be treated. As a porous material constituting the filter medium 8, there can be employed a filter cloth, wire gauze, wedge wire screen, and the like individually. There can also be employed a combination thereof.

Next there is explained a function of each assembly or memeber in an apparatus of the present invention.

In an appartus of the present invention, a vessel having inverted frustoconical configuration with a vertical central axis is effective in removing stagnant portion at the bottom of the vessel compared with a cylindrical vessel, and the like during the washing operation of powdery or granular substance, and the like wherein the agitaition of a slurry is required. A stirring assembly with spiral blade which rotates around its own axis and revolves around a central axis of the vessel is effective in improving the efficiency of mixing and reducing the stirring load for any kinds of substances such as powdery or granular substance, slurry and wet cake which have remarkably different stirring resistance one another. By disposing the above mentioned stirring assembly adjascent to the internal peripheral surface of the vessel, there can be obtained an upward motion and dispersion effect of powder or granule due to the rotation of the stirring assembly. The revolution of the stirring assembly can cause horizontal motion and at the same time remove an stagnant layer at the internal peripheral surface of the vessel. The uniformity of mixing and overall heat transfer coefficient through the sidewall are remarkably improved due to the above-mentioned rotation and revolution of the stirring assembly.

One of the features of an apparatus of the present invention is that there is provided a filter medium comprising porous material at the bottom of a vessel. The filter medium can be effectively applied to usual filtrating operation such as atomspheric filtration, pressure filtration, vacuum filtration and heating filtration, and the filter can be further effectively used as a dispersion plate to blow air in for drying in the drying operation of wet cakes of powdery or granular substance, whereby the drying efficiency can be greatly improved.

Example and Comparative Example

Next, referring to Examples and Comparative Example, effects obtained by the apparatus of the present invention is explained.

[Example 1]

An apparatus used in Example 1 comprised a fixed vessel having inverted frustoconical configuration with a vertical central axis, vertical angle of 34 degrees and a capacity of 2000 ℓ; a stirring assembly with spiral blade having a blade diameter of 250 mm and a pitch of 200 mm which was disposed adjacent to the internal peripherical surface of the vessel in such a manner that there exists clearance of 8 mm between the outer end of the spiral blade and the internal peripherical surface of the vessel; a filter medium comprising a porous plate of stainless steel (SUS-304; SUS-304 is a standard of stainless steel prescribed in JIS (Japanese Industrial Standard)) of 6 mm in thickness whereon small holes of 3 mm in diameter were made in such a manner that a pitch of holes was 6 mm and holes were arranged to form equilateral triangle, and a wire gauze of stainless steel (SUS-304) of 100 meshes piled on the porous plate.

1,500 kg of methanol slurry of polyvinyl alcohol containg 18 % by weight of solid, which is obtained by hydrolysis of polyoinyl acetate in methanol with sodium hydroxide, charged into the above-mentioned apparatus to carry out liquid separation under a pressure of 0.1 kg/cm^2G for 45 minutes.

Subsequently, 270 kg of methanol for washing was added into the apparatus and there was carried out stirring treatment for 1 minute wherein the numbers of rotation and revolution were 60 rpm and 2 rpm respectively. After the stirring treatment, a liquid separation was again carried out for 20 minutes under a pressure of 0.1 kg/cm^2G.

The temperature of a jacket was elevated up to 70°C, and nitrogen gas of 50°C was blown into the apparatus at a speed of 500 m^3N/hr through an inlet port

for drying gas disposed at the lower portion of the vessel while nitrogen gas of 50°C was blown into the apparatus at a speed of 100 m^3N/hr through an inlet port for drying gas disposed at the upper portion of the vessel in order to dry for 120 minutes under the condition that the numbers of rotation and revolution were 60 rpm and 3 rpm respectively. After drying, powder of polyvinyl alcohol was taken out from an outlet port disposed at the lower portion of the vessel for packaging.

The average particle size of obtained powder of polyvinyl alcohol was 250 μm and the content ratio of volatile matter and sodium acetate were 0.2 % and 0.9 % by weight respectively.

[Comparative Example 1]

For comparison, the same treatments as described above were carried out employing conventional process comprising liquid separation and washing operation of the above slurry using a SUPER DECANTER (horizontal type continuous decanter centrifuge with helical conveyer); transport operation of obtained wet cakes to an agitated dryer encased in jacketed through shell; drying operation of wet cakes maintaining a jacket at 70°C and blowing nitrogen gas of 50°C into the system; and transport operation of obtained powder of polyvinyl alcohol to a hopper for packaging.

The results of Example 1 and Comparative Example 1 are summarized in Table 1.

Table 1

	Example 1	Comparative Example 1
Cost ratio of plant and equipment investment	1	2
Ratio of energy efficiency	1	3
Contamination	none	present
Time for washing the system (hour)	0.5	8

[Example 2]

1,500 Kg of slurry comprising 573 Kg of crude sorbic acid, 480 Kg of ethanol and 447 Kg of water was charged into the same apparatus as in Example 1 to carry out desolving sorbic acid by heating the slurry 70°C for 30 minutes under stirring wherein the numbers of rotation and revolution of stirring assembly were 50 rpm and 4 rpm respectively.

After the complete desolution of sorbic acid, stirring was continuing and the solution was slowly cooled to room temperature so as to make sorbic acid crystallize out of the solution.

Subsequent to crystallization of sorbic acid, liquid separation was carried out for about 1.5 hrs. under a pressure of 0.1 to 0.2 Kg/cm^2G.

And then, the temperature of a jacket was elevated up to 70°C and nitrogen gas of 60°C was blown into the apparatus at a speed of 400 m^3N/hr through an inlet port for drying gas disposed at the lower portion of the vessel, while nitrogen gas of 60°C was blown into the apparatus at a speed of 100 m^3N/hr through an inlet port for drying gas disposed at the upper portion of the vessel in order to dry the wet crystal grain of sorbic acid for 4 hrs. under stirring wherein the numbers of rotation and revolution of stirring assembly were 50 rpm and 4 rpm respectively.

After drying, the crystal grain of sorbic acid was cooled to about 30°C by passing cold water through the jacket, and then, it was taken out from an outlet port disposed at the lower portion of the vessel for packaging.

543 Kg of purified sorbic acid was obstained and the content ratio of volatile matter was 0.1 % by weight.

As is obvious from the above description, there can be remarkably improved purification process comprising an operation liquid separation of powdery or granular substance, an operation of washing, an operation

of drying, and the like by way of employing an apparatus of the present invention. The apparatus of the present invention is also effective in the production process in particular wherein many kinds of chemical products are produced in a small lot since washing of a vessel can be carried out easily.

It is further understood by those skilled in the art that the foregoing description is a preferred embodiment of the disclosed device and that various changes and modifications may be made in the invention without departing from the spirit and scope thereof.

WHAT IS CLAIMED IS:

1. A treating apparatus having multifunction comprising a vessel having inverted frustoconical configuration with a vertical central axis; a stirring assembly with spiral blade disposed in the vessel adjascent to an internal peripheral surface thereof; and a driving and supporting assembly which rotates the stirring assembly around its own axis and revolves the stirring assembly around the central axis characterized in that there is disposed at the bottom of the vessel a filter medium comprising porous material.

2. The treating apparatus of claim 1, wherein a vertical angle of the vessel having inverted frustoconical configuration is 30 to 40 degrees.

3. The treating apparatus of claim 1, wherein a shape of the porous material is disc, conical or hemispheric.

4. The treating apparatus of claim 1, wherein the porous material is filter cloth, wire gauze or wedge wire screen.

FIG. 1

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