The invention relates to a method for filling apparatuses for chemical production with solids using a controllable metering device, and an apparatus, displaceable from place to place, for carrying out the method. The apparatus is suitable in particular for filling heat exchangers, absorption columns, distillation columns or tube-bundle reactors with solids.
METHOD FOR FILLING APPARATUSES WITH SOLIDS

[0001] The invention relates to a method for filling apparatuses used in chemical production with solids using a regulatable metering device, and an apparatus, displaceable from place to place, for carrying out the method. The apparatus is particularly suitable for filling heat exchangers, absorption columns, distillation columns or tube-bundle reactors with solids.

[0002] The uniform filling of solids into tubes, tube-bundles or heat exchangers is of decisive importance for the efficiency of these apparatuses. The solids may be, for example, packings, such as Raschig rings, ceramic balls or catalysts and may have different compositions and geometries, such as, for example, those of spheres, solid cylinders, hollow cylinders or rings.


[0004] U.S. Pat. No. 4,402,643 and EP 0 904 831 B1 (U.S. Pat. No. 6,170,670 B1) describe the feeding of particulate material and the associated apparatuses, the material being transported via a channel caused to vibrate into the tubes to be filled.

[0005] According to EP 0 904 831, fine dust falls through the sieve-like bottom of the channel into a separate container.

[0006] If, for example, catalyst mouldings are destroyed or damaged during filling, the resulting catalyst bed is no longer homogeneous. Fine abraded mouldings (dust) and catalyst fragments lead to the formation of cavities and channels so that nonuniform flow through the tube leads to reduced or increased pressure drops. This impairs the efficiency or throughput of the apparatus.

[0007] According to the prior art, dust is separated off by sieving but dust pollution is not avoided thereby. Dust adhering to the catalyst particles is also not separated off by this method.

[0008] This problem is solved according to the invention.

[0009] The invention relates to a method for filling tubes with solids, in which the solid is discharged from a filling funnel (2) onto a skew plane having vibrating channels (4), which is arranged in an approximately horizontal position and extends from the discharge orifice of the funnel up to at least the connection to the drop tube (5) or a flexible hose connection, through which the solid is fed to the tube to be filled, characterized in that in each case

[0010] a) the outlet tube (6) mounted on the drop tube (5) is adjusted in its height in a suitable manner,

[0011] b) dust is extracted by an extraction tube (10) and an extraction apparatus (1), which are present in the vicinity of the filling funnel (2) and outlet tube (6), and application of reduced pressure, and

[0012] c) after the filling of the tubes, the apparatus mounted on rollers or rolls (11) is moved for filling further tubes.

[0013] The solid is thus metered in a uniform manner by control or regulation of the vibration frequency of the vibrating channel (4).

[0014] The amount metered or the metering rate can also be adjusted, optionally simultaneously, by means of the flow controller (3) which is adjustable in height and has the form of a weir.

[0015] The drop tubes which are adjustable in their height or the flexible hose connections prove to be particularly advantageous because in this way small differences in the height of the tubes to be filled can also be compensated and gaps between drop tube and reactor tube are avoided.

[0016] The solids to be filled, which preferably have a mean particle size of at least 1 mm, may be mouldings of different type and suitability, such as, for example, Raschig rings, ceramic balls, inert bodies or shaped catalysts, for example in the form of granules, extrudates or pellets in the commercial dimensions.

[0017] Reactions with such catalysts are, for example, the preparation of ethylene oxide, phthalic anhydride, acrolein, acrylic acid, methyl mercaptan, hydrogen sulphide and others.

[0018] The object of the invention is to obtain a homogeneous solid bed and to avoid mechanical destruction of the solids. Zone-by-zone filling of the solids can advantageously be achieved by the method described here, so that different fillings of solids having exactly defined volume can simultaneously be introduced along the axial profile of the tube. When applied to a multiplicity of tubes (e.g. n=2-100 000), the method according to the invention is characterized by a uniform distribution of the heights of fill, so that, when the tubes are fed with a constant gas flow, differential dynamic pressures with a mean standard deviation from the mean value of in general less than 10% result.

[0019] At the same time, dust pollution should be avoided during the filling process.

[0020] According to the invention, the filling of the solid is effected by means of an apparatus as shown in FIGS. 1 and 5. With the apparatus according to the invention, x tubes (x=1-50) can be simultaneously filled. FIG. 1 shows, for example, an apparatus with which x=5 tubes can be simultaneously filled. For this purpose, the amount of solid to be filled for each reaction tube is first determined volumetrically or gravimetrically and initially introduced into a collecting vessel. The amount to be filled is preferably determined gravimetrically. The collecting vessel has at least the volume of the solids to be filled. The collecting vessel is opened and is introduced with the opening facing downwards into a filling funnel 2. The solids are loosened by means of an apparatus initially by vibrations, are caused to execute oscillatory and translational movement and are fed via a skew plane 4 (vibrating channel) uniformly to a metering device. The number of channels on the skew plane corresponds to the number of tubes to be filled simultaneously each time. The metering device is connected to the apparatus to be filled (for example a reaction tube in tube-bundle reactors) by means of drop tubes 5 adjustable in height, and permits loss-free feeding of the solids by means of an outflow tube 6 adjustable in height. Apparatuses which permit a direct connection between the apparatus for filling solid beds and the apparatus to be filled are used as the metering device. Funnels, tubes and flexible hose connections are expedient for this purpose. These metering devices are preferably designed as drop tubes (5 and 6) adjustable in
length or flexible hose connections, so that a continuous connection exists between the apparatus to be filled (for example a tube-bundle reactor) and the portioning device. As a result, the solids can be metered without scattering losses or the like into the apparatuses to be filled. Furthermore, the dust emission during the filling process is minimized. FIG. 1 shows that one connection each for an extraction apparatus 1 and an extraction tube 10, by means of which the dust optionally occurring as a result of application of reduced pressure and solid particles are extracted, is present in the vicinity of the filling funnel and the drop tubes adjustable in height.

[0021] For the filling process, it is decisive that the solid bed be fed in a uniform, slow and reproducible mass flow to the apparatus. This apparatus may be a heat exchanger, an absorption column, a distillation column and a tube-bundle reactor. Tube-bundle heat exchangers which are used as reactors are particularly preferred. These reaction tubes may also have internals (e.g. thermocouples). The apparatus according to the invention permits control of the mass flow by variation of the vibration frequency of the apparatus, advantageously by means of electrical thyristor control 7. Regulation of the mass flow is also possible by means of the flow controller 3 at the transition from the filling funnel to the vibrating channels, which flow regulator is adjustable in height. The solid bed should be fed to the apparatus by a uniform vibratory movement of the collecting container, of the skew plane and of the metering device. Advantageously, a collecting apparatus (filling funnel) for the solid filling, a feed device (skew plane) and a metering device are integrated in one apparatus. Up to 50, preferably up to 20, of these apparatuses can be integrated in a portioning device in order to fill a plurality of tubes or the like simultaneously and efficiently. FIG. 1 shows, for example, an apparatus by means of which 5 tubes can be filled simultaneously. By the simultaneous filling of a plurality of tubes, reproducible, uniform filling is ensured. For metering, up to 50 metering units (n=1–50), are expediently used in a portioning device, and from 1 to 10 metering units (X=1–10) are particularly suitable in a portioning device.

[0022] Ideally, the solids are set into motion by vibration of a skew plane. Of particular importance here is the exact and fine adjustment of the vibration in order to keep the flow rate as uniform as possible. This requirement is met by commercial electrical vibrator motors. Variation of the vibration frequency by means of an electronic control enables the intensity of vibration of the solid and hence the outflow rate thereof to be influenced. FIGS. 1 and 4 show, for example, a skew track having an arrangement of n=5 channels, which can be caused to perform vibratory movements simultaneously. This plane is advantageously closed by a preferably transparent cover (e.g. of plastic) in order to avoid a loss of solids and dust emissions.

[0023] A tube orifice 10 which, when connected to an extraction device, ensures removal of solid dust and impurities is advantageously present in the vicinity of the orifices of the metering device.

[0024] Ideally, the apparatus is mounted on rollers or rolls 11 so that it can be operated and moved simultaneously by one person. For simple handling of the apparatus, the roll can be adjusted in height.

[0025] With the aid of the method described, it is possible to keep the variation of the metering of the solids in general less than +/-5%.

EXAMPLES

Example 1

Design of the filling machines (FIGS. 1-6):

[0026] a) The filling machine has the following design:

[0027] Extraction in the upper part (1) of the filling machine and in the outlet tubes (10).

[0028] Five filling funnels (2).

[0029] Slide (3) with wing nuts at the outlet to the vibrating channel (4) for adjusting the delivery (flow controller adjustable in height).

[0030] Vibrating channel (4) via which the catalyst is transported into the drop tubes (5).

[0031] Potentiometer (7) for adjusting the intensity of the vibration of the vibrating plate.


[0033] Two handles (8, 9).

[0034] Extensions of the outlet tubes (6) with wing nuts and tapered exit which fits into the tubes of the reactor.

[0035] b) Adjustment and optimization of the filling time on the filling machines:

[0036] The filling machine is mounted above the tubes to be filled.

[0037] In general, the filling time can be adjusted by means of two parameters:

[0038] by means of the slide, acting as a flow controller adjustable in height and adjustable by means of wing nuts 3, at the outlet to the vibrating plate

[0039] by means of a potentiometer 7 which controls the vibration of the outlet channels.

[0040] It is expedient to adjust the orifice of the outlet roughly by means of the wing nut 3 and to make the fine adjustment by means of the potentiometer 7. The first fills are used for further optimization of the machine. The height of fill serves for checking the correct filling of the tubes. The machine is too fast if the tube is filled too high, or the machine is transporting the catalyst too slowly into the tube if the tube is filled too low. In both cases, the filling rate is regulated by means of the two abovementioned parameters so that the tubes are filled as homogeneously as possible.

[0041] c) Filling of the catalyst:

[0042] The solids are preferably filled zone-by-zone so that different fillings of solids are introduced simultaneously along the axial profile of the reaction tube with exactly definable heights of fill, fill volumes and masses.

[0043] Reaction tubes which contain internals for controlling the reactor are preferably filled in such a way that at least one catalyst particle per second is metered into the
tube, and the formation of dead zones and channels, which result in an increased differential pressure drop, are thus avoided.

[0044] The transport roll 11 of the filling machine is adjusted so that, after each fill of a series, the machine can conveniently be pushed forwards.

[0045] The catalyst to be filled is packed in collecting vessels, e.g. cans (1 can/tube).

[0046] The invention also relates to an apparatus, displacable from place to place, for filling tubes with solids, comprising

[0047] a) a filling funnel (2), for storing the solid,

[0048] b) a skew plane having channels (4) which are arranged thereon and which are arranged in an approximately horizontal position and extend from the discharge orifice of the funnel up to at least the connection to the drop tube (5) through which the solid is fed to the tubes to be filled, characterized in that in each case

[0049] c) the outlet tube (6) connected to the drop tube (5) can be adjusted in its height,

[0050] d) reduced pressure can be applied and dust extracted by means of an extraction tube (10) and an extraction apparatus (1) in the vicinity of the filling funnel (2) and the outlet tube (6).

[0051] e) the apparatus is mounted on rollers or rolls (11).

[0052] The cross section of the drop tube is at most equal to the cross section of the tube to be filled. Preferably, it has a smaller cross section and tapers conically at its ends so that it fits into the tube to be filled.

BRIEF DESCRIPTION OF THE FIGURES

[0053] FIG. 1 shows an arrangement according to the invention (side view) of a portioning device for the simultaneous filling of five tubes with particulate solids.

[0054] FIG. 2 shows an arrangement according to the invention of the filling funnel and of the connection for an extraction apparatus of a portioning device for the simultaneous filling of five tubes with particulate solids (view from above).

[0055] FIG. 3 shows an arrangement according to the invention of the drop tubes and of the height-adjustable outflow of a portioning device for the simultaneous filling of five tubes with particulate solids (view from the front).

[0056] FIG. 4 shows an arrangement according to the invention of the vibrating channels of a portioning device for the simultaneous filling of five tubes with particulate solids (view from above).

[0057] FIG. 5 shows an arrangement according to the invention (side view) of a portioning device for the filling of a tube with particulate solids.

[0058] FIGS. 6 shows an arrangement according to the invention (view from above) of a portioning device for the filling of a tube with particulate solids.

[0059] The numerals in the figures denote:

[0060] 1) Connection for extraction apparatus

[0061] 2) Filling funnel

[0062] 3) Flow controller adjustable in height

[0063] 4) Skew plane with vibrating channel(s)

[0064] 5) Drop tube

[0065] 6) Outflow adjustable in height (wing nut)

[0066] 7) Switchbox

[0067] 8) Carrying handles

[0068] 9) Pushing handle

[0069] 10) Extraction tube

[0070] 11) Transport roll adjustable in height

1. A method for filling tubes with solids, in which the solid is discharged from a filling funnel onto a skew plane having vibrating channels, which are arranged in an approximately horizontal position and extend from a discharge orifice of the funnel up to at least a connection to a drop tube or a flexible hose connection by which the solid is fed to the tubes to be filled, the method comprising:

- adjusting a height of an outlet tube connected to the drop tube;
- applying a reduced pressure through an extraction tube and an extraction apparatus, which are disposed in a vicinity of the filling funnel and the outlet tube, to extract dust; and
- moving the apparatus mounted on rolls or rollers, after the filling of the tubes, for filling further tubes.

2. The method according to claim 1, wherein the solids have a geometry of spheres, solid or hollow cylinders, stars or rings.

3. The method according to claim 1, wherein tubular or plate-type heat exchangers, which are used as reactors, are filled.

4. The method according to claim 1, wherein an amount of solid to be filled is determined for apparatuses initially by volumetric or gravimetric determination and is fed to the filling funnels of a portioning device in loose form or in collecting vessels remaining in the filling funnels.

5. The method according to claim 1, wherein a plurality of apparatuses are filled in parallel simultaneously.

6. The method according to claim 1, wherein a feed device is designed so that the funnels containing the solids empty onto the skew plane which has parallel passages as the channels in the number of the tubes to be filled in each case and is closed by a cover.

7. The method according to claim 1, wherein the solids are filled zone-by-zone and different fillings of solids are introduced simultaneously along an axial profile of a reaction tube with predetermined heights of fill, fill volumes and masses.

8. The method according to claim 1, wherein reaction tubes which contain internals for controlling a reactor are filled such that at least one catalyst particle per second is metered into the tube.

9. The method according to claim 8, wherein an amount metered of the solids to be filled is adjusted by a flow controller which is adjustable in height.
10. The method according to claim 1, wherein a portioning device is mounted on rollers or rolls which are adjustable in height to be operated and moved simultaneously by one person.

11. An apparatus, displaceable from place to place, for filling tubes with solids, comprising
   a filling funnel configured to store the solid;
   a skew plane comprising vibrating channels arranged in an approximately horizontal position and extending from a discharge orifice of the funnel up to at least a connection to a drop tube or a flexible hose connection by which the solid is fed to the tubes to be filled, wherein in each case
   an outlet tube is connected to the drop tube and is configured to be adjusted in height,
   reduced pressure is applied and dust is extracted through an extraction tube and an extraction apparatus in the vicinity of the filling funnel and the outlet tube, and
   the apparatus is mounted on rollers or rolls.

12. The apparatus according to claim 11, wherein a cross section of the outlet tube is at most equal to that of the tube to be filled.

13. The apparatus according to claim 11, wherein the outlet tube tapers conically at an end and the end has a smaller cross section than the tube to be filled.

14. The apparatus according to claim 11, wherein the skew plane with the vibrating channels is closed by a cover.

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