(21) Application No. 2857/78

(22) Filed 24 Jan. 1978

(31) Convention Application No. 2702693 (32) Filed 24 Jan. 1977 in

(33) Fed. Rep. of Germany (DE)

(44) Complete Specification published 26 Aug. 1981

(51) INT. CL.3 B01J 1/00

(52) Index at acceptance

B1F 4E1



(71)We, FRIED. KRUPP GESSELL-SCHAFT MIT BESCHRÄNKTER HAFTUNG, of 103 Altendorfer Strasse, D-4300 Essen 1, Federal Republic of Germany, a German Body Corporate, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described

in and by the following statement:-10 It is known that chemical and/or physical processes, in particular thermal processes, can be carried out in a dust cloud. A dust cloud consists of a gas, constituted by one or more gaseous substances, and particles of one or more solid materials suspended in the flowing gas. The dust cloud is in movement and flows through a reaction zone, in which a chemical reaction, or a physical operation, such as a heat exchange operation, an adsorption operation or classification of solids takes place. The gas serves as a transport medium for the solid particles but it can also take part in the process taking place in the dust cloud. After the dust cloud has flowed through the reaction zone it is separated into its constituents in a separator, for example a dust removing chamber or a cyclone. Gas and solid particles leave the separator by different paths and are either passed on for further use, partially rejected as an unusable reaction product or returned to the dust cloud. In general the process is not quantitative with a single passage of the dust cloud through the reaction zone so that at least

particles is necessary. The dust cloud is formed by mixing a gas stream with solid particles of suitable size. A dust cloud is to be distinguished from a fluidised bed because the gas and all the solid particles in a dust cloud flow continuously in the same direction and at substantially the same speed through any given zone, while in a fluidised bed the bulk of the solid particles remain suspended in the bed and only the gas flows through the bed carrying some solid particles with it.

partial recycling of the gas and/or the solid

It is also known, after separation of the dust cloud, to convey the solid particles from the separator mechanically by swinging flaps, worm conveyors, paddle wheels and like devices and to return them if required to the dust cloud. All these forms of discharge apparatus, however, have the disadvantage that they include moving parts subject to 55 wear, which involves proneness to disturbance and high energy and maintainence

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The object of the invention is to provide a method and apparatus for carrying out chemical and/or physical processes in a dust cloud which operates with certainty and at favourable cost, makes possible a uniform concentration of solids in the dust cloud and can be readily adapted to the requirements of 65 the particular process to be carried out.

The invention is based on the discovery that the solid particles separated in the separator can be removed by free fall into a column of particulate solid material, which is 70 variable in height, and contained in a vessel into the lower end of which a stream of gas is introduced to form the dust cloud, the height of the column controlling the solids content of the dust cloud and being sufficient to form 75 a seal to prevent gas from flowing from the vessel to the separator. Provided the flow resistance of the column exceeds the sum of the flow resistances of the reaction zone, the separator and other components of the apparatus through which the gas passes, no gas will flow from the column directly to the separator and a dust cloud will flow from the vessel into the reaction zone. As the height of the column is increased, the concentration of 85 solids in the dust cloud increases.

The invention accordingly provides a method of carrying out a chemical and/or physical process which comprises forming a dust cloud by feeding a stream of gas into the lower end of a vessel containing a column of particulate solid material of determined but variable height sufficient to prevent the gas from flowing through the column, passing the gas laden with solid particles and forming 95 the dust cloud from the vessel through a reaction zone and then through a separator which separates it into gaseous and solid constituents and replenishing the column to maintain it at its determined height by 100

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addition to it of fresh solid particles and/or solid particles separated from the dust cloud in the separator. Preferably the replenishing particles are fed by gravity to the column through a dip tube.

The method can easily be adjusted to different process conditions by variation in the height of the column and ensures a certain, uniform and cost-favourable course of the process, in particular in the case of high temperature processes. The method can be carried out under suction or under pressure and can take place in several stages in series.

The separated solid particles may be returned to the column until the chemical and/or physical process between solids and gas is ended. This has the advantage that they can operate in mutual relationship until a limiting condition in terms of reactionability is attained.

The invention includes an apparatus for carrying out the above method comprising a vessel containing the column, a reaction tube having an inlet connected to the vessel, a separator connected to the outlet of the reaction tube, a nozzle for introducing the gas into the lower end of the vessel, and a dip tube in the upper end of the vessel which is adjustable in level and is connected to receive by gravity separated solid particles from the separator and fresh solid particles from a supply bunker. This apparatus involves no mechanically moved parts. 35

At least one reaction chamber may be connected between the reaction tube and the separator, which may consist of at least one cyclone. It is advantageous to provide the reaction chamber and/or the separator with dip tubes which are adjustable in level. This provides additional control of the course of the process. The solids content of the dust cloud in the reaction zone is independent of throughput and a desired loading can be achieved. The range of control is between about 0.001 and 60 volume %, preferably between 0.1 and 20 volume %, of solids. By the use of at least one reaction chamber in addition to the reaction tube a longer dwell time of the dust cloud in the reaction zone is achieved.

When carrying out processes involving a high working temperature it is advantageous for all parts of the apparatus to be lined with refractory material, for example fire clay bricks and refractory materials based on silica, alumina, magnetite and/or chromium oxide. When carrying out processes under high or subatmospheric pressures the parts of the apparatus are suitably dimensioned and sealed.

In particular the invention permits of successful operation of the following chemical and/or physical processes:-

(a) Waste gas purification. Removal of

substances such as HF, SO<sub>2</sub>, NO<sub>3</sub>, HCl, H<sub>2</sub>SO<sub>4</sub> by basic substances such as CaO, Ca(OH)<sub>2</sub>, Na(OH) from waste gases from metallurgical and chemical plants.

(b) Iron recovery. Direct reduction of 70 iron ores, especially with CO and/or H2 as

reducing gas.

(c) Drying of gases. Adsorption of gaseous solvent residues or water by active carbon or molecular sieve.

(d) Thermo-chemical processes. Burning and calcining, e.g. for production of lime and cement, as well as gasification of coal with optional desulphurization, e.g. by addition of lime.

The invention will now be further explained by reference to the drawing.

A dust cloud is fed, from a dust cloud forming vessel 5 containing a dip tube 8, by a pipe 12 into a reaction zone, which consists of a reaction tube 2 and a funnel-shaped reaction chamber 3 which are interconected by a pipe 13. In the reaction zone a chemical and/or physical process takes place within the dust cloud. If desired, heat is supplied to or withdrawn from the reaction tube 2. The dwell time and solids content of the dust cloud can be controlled by raising and lowering a dip tube 10. Thus raising the dip tube 10 increases the effective volume of the 95 chamber 3, the dwell time of the dust cloud in the chamber and the pressure loss in the chamber. This increase in pressure loss lowers the solids content of the dust cloud in the tube 2 and also in the chamber 3. The dust 100 cloud leaves the reaction chamber 3 through a pipe 14, in which the reaction may continue, to a cyclone separator 4, in which the dust cloud is separated into its gaseous and solid constituents. The time and efficiency of 105 separation can be controlled within limits by variation in level of a dip tube 11. Solid particles are removed from the separator 4 by a pipe 26. The gas, largely freed from solids, is fed through a pipe 15 to a filter 6, where 110 the remaining solid particles are removed quantitatively. The gas leaving the filter 6 is discharged to atmosphere through a pipe 16 or recycled, wholly or partially, to the process through a pipe 17. Fresh gas can be supplied 115 as required through a pipe 18 discharging into the pipe 17. The gas is compressed in a compressor 19 and fed through a pipe 20 to a main nozzle 9 in the lower part of the vessel 5. The gas flowing through the nozzle 9 sucks 120 in solid particles from a column 22 of particles in the vessel 5 to form the dust cloud which flows through the pipe 12 to the reaction zone. Near the nozzle 9 are loosening up nozzles 21, through which a weak gas 125 flow 21 enters the vessel 5 to loosen up the solid particles near the nozzle and bring them into condition ready to flow. The solid particles collected in the filter 6 can be rejected through a pipe 27 or returned to the 130

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vessel 5 through a pipe 24. The whole or part of the solid particles separated in the separator 4 falls freely into the vessel 5 through a pipe 25 and the dip tube 8. A chain-dotted line 28 indicates an alternative recycling path for the solid particles, to which the pipe 27 can also be connected.

The height of the column 22 is such that its weight exerts sufficient pressure on the gas entering through the nozzle 9 to prevent it from passing through the column and the dip tube 8. The gas takes the path of least resistance and picks up some of the solid particles to emerge as a dust cloud through the pipe 12. The solids content of the dust cloud depends on the height of the column 22.

When no solids are withdrawn through the pipes 26, 27 and all the solid particles separated in the separator 4 and the filter 6 are returned to the column 22 an equilibrium prevails in the system. As soon, however, as solids are removed through the pipe 26 or 27, the level of the column 22 will fall below the mouth of the dip tube 8 and fresh solid particles will be supplied to the column from a bunker 1, through a pipe 23 and the dip tube 8, so restoring the column 22 to the height at which it again blocks the mouth of the dip tube 8.

The height of the column 22 can, of course, be adjusted by raising or lowering the dip tube 8.

The use of the apparatus for defluorination of waste gas will now be described.

A waste gas containing about 100 mg HF/Nm3 and flowing at a rate of 3000 Nm<sup>3</sup>/h and at a temperature of 80°C, was introduced through the nozzle 9 into the vessel 5. The bunker I contains softly burnt lime (CaO) of particle size about 4  $\mu m$  to 3 mm, which was fed through a pipe 23 and the dip tube 8 into the vessel 5. A column 22 of particulate material 80 mm in height was formed in the vessel 5. The reaction zone included two funnel shaped reaction chambers 3 as well as the reaction tube 2 and had a total length of about 20 m. The reaction tube 2 had a diameter of about 200 mm and the pressure drop in the reaction zone was about 400 mm water column. The solid particles separated in the cyclone 4 were returned to the container 5 through the pipe 25. The degree of separation of the cyclone was set to 80%. The gas leaving the cyclone 4 and containing the rest of the solid particles was divided by a Y piece into two streams (pipes 24 and 27). One stream (pipe 24) was returned to the process and the other stream (pipe 27) was discharged in a quantity corresponding to the amount of lime newly

supplied to the vessel 5 through the dip tube

8. The added amount of lime was so adjusted

that the maximum amount of CaF<sub>2</sub> in the circulating solid material did not exceed 30%.

The solid content was controlled by taking samples and chemical analysis. The purified gas discharged to atmosphere from the filter contained, at maximum, 0.7 mg F/Nm3.

## WHAT WE CLAIM IS:—

1. A method of carrying out a chemical and/or physical process which comprises forming a dust cloud by feeding a stream of gas into the lower end of a vessel containing a column of particulate solid material of determined but variable height sufficient to prevent the gas from flowing through the column, passing the gas laden with solid particles and forming the dust cloud from the vessel through a reaction zone and then through a separator which separates it into gaseous and solid constituents and replenishing the column to maintain it at its determined height by addition to it of fresh solid particles and/or solid particles separated from the dust cloud in the separator.

2. A method according to claim 1, wherein the replenishing particles are fed by gravity to the column through a dip tube.

3. Apparatus for carrying out the method according to claim 2, comprising a vessel containing the column, a reaction tube having an inlet connected to the vessel, a separator connected to the outlet of the reaction tube, a nozzle for introducing the gas into the lower end of the vessel, and a dip tube in the upper end of the vessel which is adjustable in level and is connected to receive by gravity separated solid particles 100 from the separator and fresh solid particles from a supply bunker.

4. Apparatus according to claim 3, which includes at least one reaction chamber connected between the reaction tube and the 105

5. Apparatus according to claim 4, in which the reaction chamber is funnel shaped.

6. Apparatus according to claim 4 or claim 5, in which the reaction chamber is 110 provided with a dip tube which is adjustable in level.

7. Apparatus according to any one of claims 3 to 6, in which the separator consists of at least one cyclone.

8. Apparatus according to claim 7, in which each cyclone is provided with a dip tube which is adjustable in level.

9. Apparatus according to any one of claims 3 to 8, in which the vessel contains 120 lossening nozzles near the nozzle which admits the gas forming the dust cloud.

10. Apparatus according to any one of claims 3 to 9, in which all parts are lined with refractory material.

11. Apparatus according to claim 3, substantially as described herein with reference to the accompanying drawing.

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Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon) Ltd. —1981. Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

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1 SHEET COMPLETE SPECIFICATION
This drawing is a reproduction of the Original on a reduced scale

