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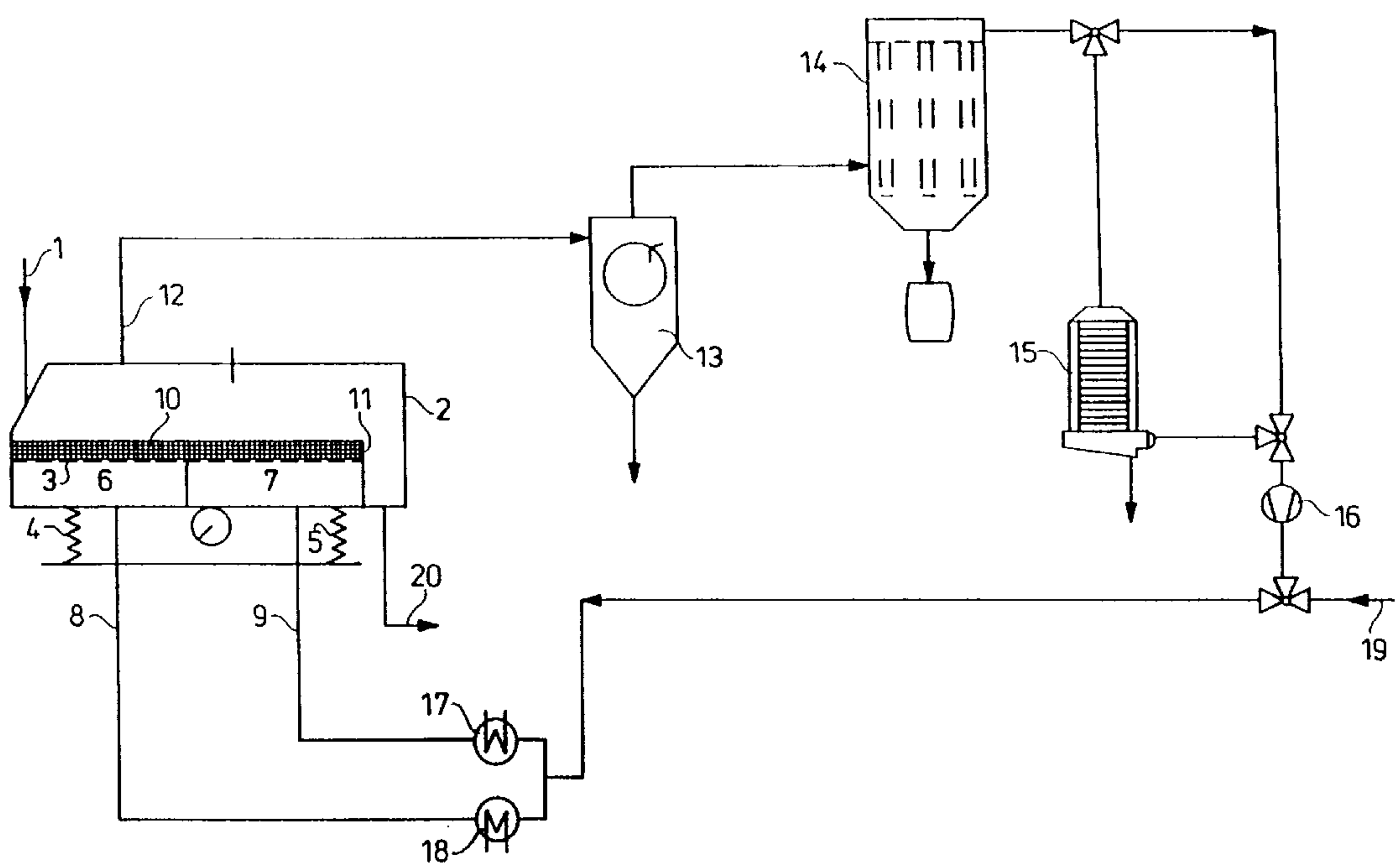
(54) **PROCEDE DE DEGAZAGE DE CAOUTCHOUC OBTENU PAR POLYMERISATION EN PHASE GAZEUSE**

(54) **PROCESS FOR DEGASSING RUBBER FROM GASEOUS PHASE POLYMERISATION**



(57) L'invention concerne un procédé pour dégazer des particules de caoutchouc obtenues par polymérisation en phase gazeuse. Selon ce procédé, les particules de caoutchouc sont maintenues constamment en mouvement dans un lit fluidisé à vibrations tout en étant traversées par un flux de gaz.

(57) In a process for degassing rubber particles from gaseous phase polymerisation, the rubber particles are constantly held in motion in a vibrating fluidised bed and are crossed at the same time by a gas flow.

(57) Abstract

In a process for degassing rubber particles from gaseous phase polymerisation, the rubber particles are constantly held in motion in a vibrating fluidised bed and are crossed at the same time by a gas flow.

Process for degassing rubber from gaseous phase polymerisation

The present invention relates to a process for degassing rubber particles from gaseous phase polymerisation, in which the rubber particles are kept in constant motion in a vibrating fluidised bed while a gas stream is simultaneously passed through them.

Polymerisation of polyolefins in the gaseous phase is a process which has long been known and which was first carried out on an industrial scale in 1968 (Ullmanns Encyclopedia of Industrial Chemistry, 4th edition 1980, Vol. 19, p. 186 ff).

In this process, the polymerisation reaction proper takes place in a fluidised bed reactor, which consists of a reaction zone and a superposed stabilising zone, in which the solid particles are extensively separated from the gaseous phase. The monomers, additives and catalyst are introduced into the reaction zone. To maintain a fluidised bed, a circulating gas stream is fed to the reactor from below. This circulating gas stream, which consists substantially of the unreacted monomers, is drawn off again at the top of the reactor, freed from residual particles, cooled and returned to the reactor. The polymer arising is drawn off continuously or semi-continuously from the reaction zone and subjected to further processing.

This further processing comprises, inter alia, the degassing of the polymer particles produced, because current requirements with regard to the purity of the polymer particles are high.

During degassing, the residual monomers and other impurities which exhibit adequate vapour pressure at the corresponding temperatures are stripped from the tacky polymers produced by gaseous phase polymerisation.

The tackiness of the rubber particles in particular makes them difficult to degas. This tackiness of the rubber particles leads to particle agglomeration and thus to a reduced material exchange surface area. In addition, the rubber particles also clog up the apparatuses, thereby shortening their service life.

The residual monomers are currently removed in extruders, for example, which are either under vacuum or have a carrier gas passing through them. This process exhibits the disadvantage either of being incapable of reducing the residual monomer content of the polymer particles sufficiently or of requiring very complex equipment. Another process entails the removal of monomers in a solid bed through which carrier gas is passed. In the case of rubber, this may rapidly lead to lump formation, which is accompanied by a poor degassing outcome and problematic discharge.

The object is therefore to provide a process for degassing rubbers from gaseous phase polymerisation which does not exhibit the disadvantages of the prior art.

The object is achieved according to the invention by the provision of a process for degassing rubber from gaseous phase polymerisation, in which the rubber particles are kept in constant motion in a vibrating fluidised bed while a gas stream is simultaneously passed through them.

The process according to the invention is preferably carried out in an apparatus which consists principally of a vibrating base plate through which gas may flow. This base plate is preferably horizontally disposed and is preferably rectangular, any other shape also being possible. The base plate may take the form of a perforated plate, a slotted plate or a Conidur plate or exhibit any other air-permeable geometry. Perforated plates are preferred, however. The holes preferably have a diameter of from 1 to 4 mm. The distribution and spacing of the holes is such that an open surface area of from 1 to 6 %, preferably 1.3 to 4 %, is obtained.

According to the invention, the base plate must vibrate preferably at a frequency of 200 to 2000 min^{-1}, particularly preferably 1400 to 1600 min^{-1}. The amplitude should preferably amount to 1 to 25 mm, particularly preferably to 3 to 10 mm.

The polymer particles to be degassed are located on the base plate, wherein the vibrating fluidised bed should not exceed a maximum height of 200 mm. The height of the vibrating fluidised bed preferably amounts to from 80 to 150 mm.

A gas, preferably heated to 60 to 120 °C, is passed through the base plate and the polymer particles located thereon. The gas is preferably industrial N_2. However, the gas stream may also preferably consist of a mixture of N_2 and steam or pure steam. The steam is preferably superheated.

The gas loading rate of the base plate amounts preferably to 1500-4000 m^3/m^2h.

Degassing of the rubber particles may be carried out both continuously and discontinuously. The residence time of the rubber particles on the base plate is to be determined in accordance with the permissible content of residual monomers in the rubber particles. However, it preferably amounts to between 2 minutes and half an hour.

In a preferred embodiment, the rubber particles and the gas stream are conveyed in a cross flow relative to each other. This is achieved in that the rubber particles are fed in on one side of the base plate and drawn off again on the opposite side, while the gas is passed through them from below. This method of operation has the advantage that conveying of the rubber particles continues during degassing.

The gas stream used for degassing is preferably circulated.

The process according to the invention may be used to degas polymer particles of any type produced in the gaseous phase, but it may be used particularly preferably to degas rubber particles of any type.

For the purposes of the invention, polymers comprise, for example, poly-α-olefins, polyisoprene, polystyrene, SBR, IIR, polyisobutene, polychloroprene, silicones and copolymers of ethylene and one or more of the following compounds: acrylonitrile, malonic acid esters, vinyl acetate, acrylic and methacrylic acid esters, vinyl acetate, acrylic and methacrylic acid esters, α-olefins, dienes and trienes.

For the purposes of the invention, rubbers comprise uncross-linked, but cross-linkable, polymers, which may be rendered rubbery by cross-linking.

However, the process according to the invention is preferably used in the gaseous phase polymerisation of EPM, EPDM, SBR, NBR, polyisoprene and BR.

The polymer particles are preferably spherical in form, with a diameter preferably of 1-7 mm.

The invention will be described below with reference to **Figure 1**.

Fig. 1 shows the process according to the invention with gas circulation.

Figure 1 illustrates degassing according to the invention. The rubber particles contaminated with residual monomers, which were produced in a gaseous phase reaction, are fed continuously into the vibrating channel fluidised bed drier 2 on the left-hand side 1 thereof. This drier principally consists of a perforated base plate 3, which is mounted so as to vibrate and in this example is caused to vibrate by an unbalance motor. The perforated base plate is acted upon from below by a hot gas stream via two chambers 6 and 7, which are in turn supplied by the lines 8 and 9. Alternatively, the chamber 7 may be acted upon by cold gas for cooling purposes.

Once the contaminated rubber particles have been fed in metered manner through the inlet 1 onto the vibrating base plate of the vibrating channel fluidised bed drier 2, said base plate is caused to vibrate. At the same time, gas is passed through it from below, such that the fluidised layer 10 forms. In this layer, the rubber particles are degassed and additionally conveyed from left to right, where, free or virtually free of residual monomers, it leaves the apparatus via the outlet 20. This manner of operation allows the rubber particles and gas stream to travel in cross flow relative to each other. The height of the vibrating fluidised bed 10 on the base plate 3 is determined by the height of the weir 11. Alternatively, a weir may be omitted, but then the angle of inclination of the apparatus must be such that the desired residence time is achieved.

The gas stream containing residual monomer is drawn off at the top of the apparatus via the lines 12 and first of all cleaned of polymer particles entrained by the gas stream. This cleaning is carried out by means of a cyclone 13 and a filter 14. Then, at least some of the gas stream is passed through a condenser, to condense out the residual monomers at least partially. The gas stream cleaned in this way is then compressed using the fan 16, before being heated by the heat exchangers 17 and 18 and then recycled in the vibrating channel fluidised bed drier 2. Additional gas may optionally be supplied in metered manner via the connection 19.

Division of the gas stream into two lines 8 and 9, each comprising a heat exchanger 18 and 17, allows it to be supplied to the fluidised layer 10 at different temperatures.

The process according to the invention allows the obtainment of very low residual monomer contents in the rubber particles, without damaging the product. Long-term tests have shown that the apparatuses used do not become clogged and thus exhibit a long service life with low expenditure on maintenance. Moreover, the process according to the invention is comparatively favourable.

Example:

368 g of a polybutadiene rubber produced in the gaseous phase and having a residual butadiene content of 1.4 % was degassed in a vibrating batch fluidised bed drier with nitrogen at 80 °C. The volumetric flow rate was 30 Norm-m^3/h. The fluidised bed drier, made by Vibra/Offenbach (D), had a fluidisable base area of 0.15 m x 0.15 m. The distributor base plate in the form of a perforated plate comprised holes with a diameter of 2.5 mm, in a triangular arrangement at a spacing of 19.5 mm, such that an open surface area of 1.3 % was obtained. The vibrations exhibited a frequency of 1490 min^{-1} at an amplitude of 2.5 mm. In the fluidised state, the height of the rubber was approximately 40 mm, which corresponds to a bed density of 400 kg/m^3.

After a residence time of 5 mins, it was possible to reduce the residual butadiene content in the polymer particles to 60 ppm. After a residence time of 10 mins, the residual butadiene content was below 10 ppm.

Claims

1. A process for degassing polymer particles, in particular rubber particles, from gaseous phase polymerisation, characterised in that the polymer particles are kept in constant motion in a vibrating fluidised bed while a gas stream is simultaneously passed through them.

2. A process according to claim 1, characterised in that the vibrating fluidised bed is produced by means of a vibrating base plate through which gas may flow.

3. A process according to one of claims 1 or 2, characterised in that the base plate vibrates at 200 min^{-1} to 2000 min^{-1}, preferably at 1400 min^{-1} to 1600 min^{-1} and in that the amplitude amounts to from 1 to 25 mm, preferably 3 - 10 mm.

4. A process according to one of claims 1 to 3, characterised in that the gas stream is a nitrogen stream or a nitrogen/steam stream or a pure steam stream and exhibits a temperature of from 60 °C to 120 °C.

5. A process according to one of claims 1 to 4, characterised in that the rubber particles and the gas stream are conveyed in a cross flow relative to each other.

6. A process according to one of claims 1 to 5, characterised in that the gas stream is circulated.

7. A process according to one of claims 1 to 6, characterised in that the rubber particles are EPM, EPDM and BR.

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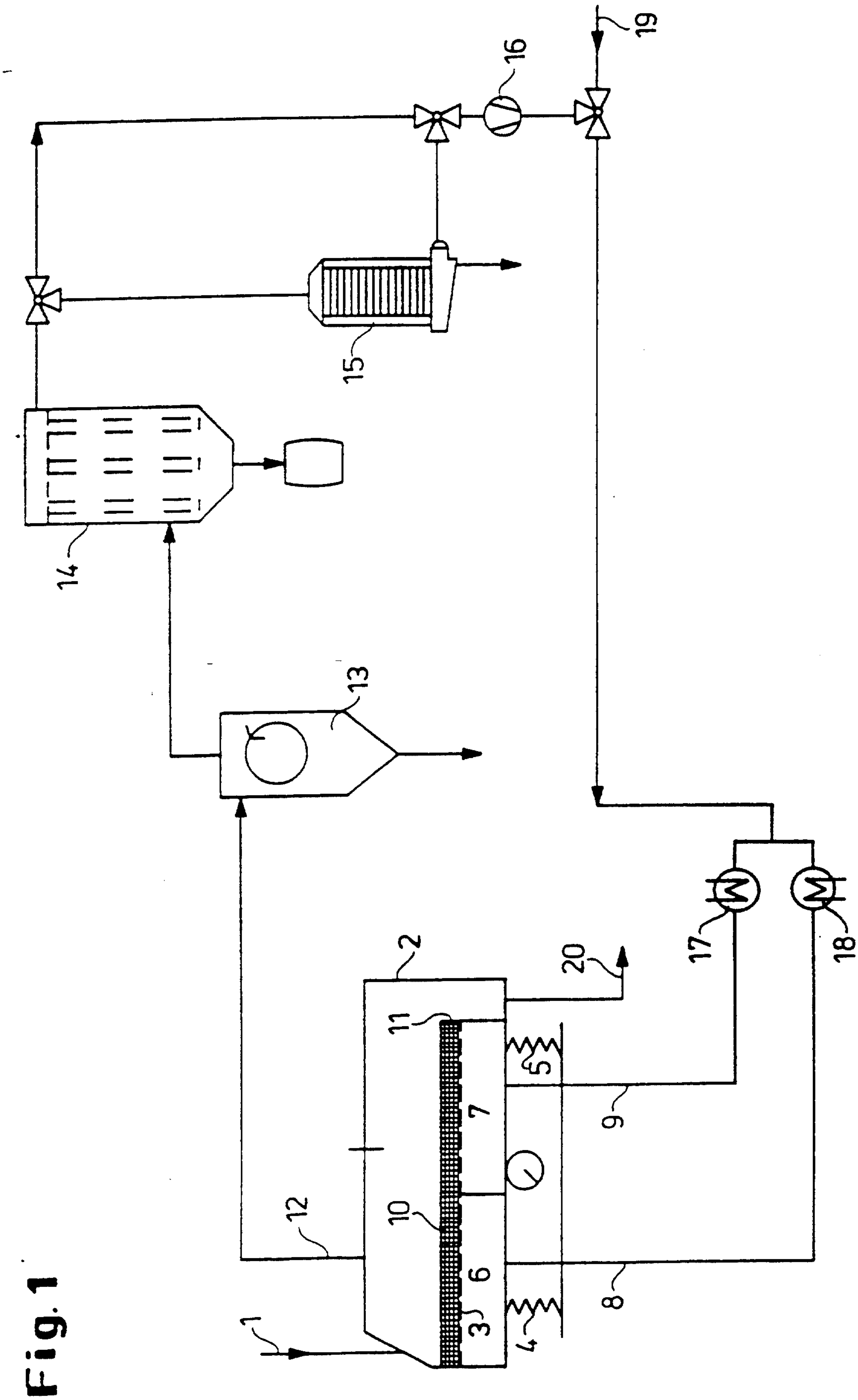

Fig.1