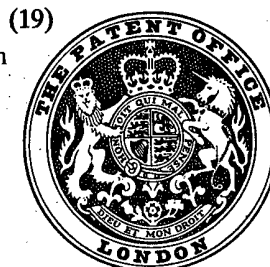


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(72) Inventors: CLEMENS CASPER
 AXEL LIPPERT
 JOHANNES OTTO SAJBEN



(54) MULTIPHASE FLOW TUBE FOR MIXING, REACTING
 AND/OR EVAPORATING COMPONENTS

(71) We, BAYER AKTIENGESELLSCHAFT, a body corporate organised under the laws of Germany, of 509 Leverkusen, Germany; 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:

This invention relates to an apparatus for mixing, reacting and/or evaporating components consisting of a helically wound tube having a feed means projecting through the closed inlet end of the tube and a separation vessel at the outlet end of the tube, the coiled tube being surrounded by a tempering jacket divided into sections.

In process engineering, substances are frequently separated or mixed. At the same time, reactions may occur. Particular difficulties are presented by substances which, in order to obtain a satisfactory quality, may only be treated in a narrow temperature range under certain pressure conditions. The treatment is particularly critical in the case of substances of high viscosity because the residual component becomes increasingly more difficult to evaporate with falling vapour pressure. In many cases, undesirable, barely resolvable substance concentrations occur even when substance components are mixed in and may give rise to reductions in the quality of the product, particularly in the case of exothermic reactions.

There are a whole number of types of apparatus which are used for mixing and evaporating and, in some cases, also for reactions.

Thus, a screw evaporator is known in

which one or more heatable screws in a housing provide for the transport and, optionally, mixing of the liquid mixture, the component to be evaporated off optionally being run off *in vacuo* from the empty overlying space.

The high viscosity of the liquid layer and the small evaporation surface complicate evaporation. The construction is complicated and unreliable in operation on account of the presence of rotating parts. The installation and operating costs are high.

In thin-layer evaporation, a liquid film from which one of the components evaporates is produced by mechanically rotating wiper elements on the heated wall of an elongate, cylindrical container.

Evaporation of the residual component decreases continuously with falling vapour pressure unless a vacuum is applied. However, vacuum increases the danger of undesirable foaming. The rotating elements increase unreliability in operation. The investment and operating costs are high.

In falling-film evaporators, there are several parallel vertical tubes along whose inner wall liquid flows down in a thin layer, whilst gas flows through in the free inner space in the same direction or in the opposite direction. On account of the limited tube length, the residence time is often not long enough for residual evaporation, especially since a certain quantity has to flow through in order to prevent local overheating by temporary breakage of the film which may lead to destruction of the heat-sensitive material. In addition, it is only possible to use liquids of low viscosity in whose case the undesirable entrainment

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of liquid droplets cannot always be avoided.

5 In tubular coil evaporators, a polymer
solution is heated under excess pressure and
is expanded through a nozzle into a helically
wound tube, a liquid film being driven along
the wall by the internally flowing gas stream
to form a secondary stream. One disadvan-
10 tage of this apparatus is that it is not possible
to influence an operation once it has started.
There is nothing flexible in the mode of
operation of the apparatus. The specific
output decreases towards the tube exit.
When vacuum is applied, the output limit is
15 rapidly reached by the critical mass flow
density which determines the moment at
which the film breaks. The apparatus may
only ever be used for evaporating a particu-
lar substance component.

20 Accordingly, an object of the present
invention is to provide an apparatus which,
under a predetermined pressure and at a
temperature controlled in sections over a
wide working range, provides either for
intensive mixing by the measured addition
25 of substance components to a liquid, option-
ally with simultaneous control of a certain
reaction sequence, and/or for a thermal
separation of one or more substance compo-
nents, optionally with recovery of individual
30 fractions, the substances being carefully
treated, even in the case of viscous liquids,
with high selectivity in a static apparatus.

According to the present invention, this
object is achieved by virtue of the fact that
35 one or more enclosed conduits extend later-
ally from the tube and project into the
interior of the tube. When more than one
conduit is present, these conduits are
arranged along the axis of the coiled tube.

40 The present invention therefore relates to
an apparatus for mixing, reacting and/or
evaporating components which comprises a
helically wound tube, a feed means project-
ing through the closed inlet end of the tube
45 and a separation vessel at the outlet end of
the tube, the coiled tube being surrounded
by a tempering jacket divided into sections
and one or more enclosed conduits extend-
ing laterally from the tube, projecting into
50 the interior of the tube and, when more than
one conduit is present, being arranged along
the axis of the coiled tube.

55 The advantages afforded by the present
invention are the fact that the intensive
process of a multiphase flow tube may be
influenced from outside by built-in conduits.
Thus, it is possible to introduce identical or
different substance components at certain
60 points of the coiled tube in such a way that
the mixture is able largely to homogenise
again before any new addition is made. In
this way, harmful concentrations of subst-
ances are avoided. The measured addition is
65 particularly important in the case of ex-
othermic or endothermic reactions in which

product damage is caused by the occasional-
ly very narrow temperature range being
exceeded.

The partial pressure may also be kept low
by the addition of gas, particularly inert gas,
70 so that evaporation remains substantially
constant over the entire length of the tube.

On the other hand, it is also possible to
run off components. Evaporation is facili-
tated by the drawing off of vapours. The flow
75 rate is reduced so that the film of liquid does
not break up on the wall.

In addition, it is possible to draw off gas
or liquid in fractions, the process being
assisted by a heating medium which may be
80 present in the tempering jacket divided into
sections.

The apparatus is extremely flexible in use.
In addition, it is possible to cover all
working ranges by the position of the
85 conduits and by the length of the coiled
tube.

Despite the careful treatment of the
products, the specific output is high.

90 The apparatus is compact, simple in
structure and easy to maintain. Due to the
absence of rotating parts, it is reliable in
operation. The investment and operating
costs are low.

In one particular embodiment, the feed
95 means is in the form of a two-component
nozzle. As used herein the term "two-
component nozzle" means that the nozzle is
provided with two separated subsidiary noz-
zles both of which can introduce a compo-
100 nent into the apparatus.

Generally, a large amount of gas is
required for driving the liquid ring along the
coiled tube. In general, a heated liquid is
introduced by means of a nozzle, and after
105 expansion, steam is produced which drives
the liquid ring. It is also possible, however,
to use a two-component nozzle by which
inert gas in particular may be added as the
second component to drive the liquid ring.
110 The advantage of this is that, despite the
lower vapour pressure of the component to
be evaporated, it avoids the need for
vacuum which would involve the danger of
foaming.

115 In another embodiment, the end(s) of the
conduit(s) in the coiled tube is (are) in the
form of a nozzle(s).

Depending upon the arrangement and
configuration thereof, the nozzles enable
120 the component to be added to be so finely
dispersed that intensive mixing is obtained.

In another embodiment, the end(s) of the
conduit(s) in the coiled tube is (are) in the
form of opening(s) for the discharge of
125 fluid.

Depending upon the configuration of the
vents and the position of the openings
thereof relative to the axis of the coiled
130 tube, it is possible either to draw off the

vapours flowing inside the tube or to strip off part of the liquid ring at the edge. In this connection, it is important that the disruption of the liquid film should be minimised by stream-lining the fittings.

In one particular embodiment, the or each conduit is connected to a metering device.

Through one or more metering devices, the individual conduits may be charged with certain quantities of the components to be added in such a way that the mixing or reaction process takes place in stages according to programme. The evaporation process may also be improved by adding gas or externally generated steam to lower the partial pressure of the component to be evaporated to such an extent that there is no need for an expensive vacuum to be applied.

In one useful embodiment, the or each conduit is connected to an evacuation system.

As stated above, it is possible to obtain a phase separation in the multiphase flow tube, with vapours flowing inside the tube and a liquid ring being present at the edge of the tube. This selective separation of the phases in the multiphase flow tube enables one phase to be drawn off without anything of the other phases being entrained. In this way, it is possible to draw off part of the medium and, hence, to reduce flow rate before the critical mass flow density is reached at which the liquid film is broken up by the internally flowing gaseous medium and hence leads to product damage by overheating. This is of particular advantage with very high vacuums (≤ 10 Torr) or with large evaporation volumes.

In one embodiment, the tube diameter of the coiled tube decreases towards the tube exit.

By tapering the tube, the wetting surface is made smaller so that, even with a high vapour component, the film is prevented from breaking up and causing product damage by the resultant overheating of the wall surfaces.

Some embodiments of the present invention are illustrated in the following with reference to the accompanying drawings, wherein:

Figure 1 is a section through a two-phase flow tube.

Figure 2 is a section through a nozzle.

Figure 3 is a section through a pipe-shaped outlet conduit.

Figure 4 is a section through an intake conduit having a slotted cross-section.

Figure 5 is a section through a stripper.

Figure 1 shows a coiled tube 1, with lateral conduits 2, including valves 3, and a tempering jacket, 4, which is divided into sections.

A feed means, 6, which in this case, is in

the form of a two-component nozzle, 7, projects through the closed inlet end, 5, of the coiled tube 1. The coiled tube, 1, terminates at its outlet end, 8, in the separation vessel, 9, which has a vapour outlet, 10, and a liquid outlet, 11.

Figure 2 shows an inlet nozzle, 12, at the end of the conduit, 2, which uniformly disperses one or more substance components in the gaseous stream.

Figure 3 shows a pipe-shaped vent, 13, at the end of the conduit, 2, which by virtue of its shaping, prevents liquid from flowing along the conduit to its opening in order to be entrained there during removal of the vapour.

In Figure 4, a conduit, 2, is in the form of an aerodynamically rounded conduit, 14, having a slotted cross-section which is particularly suitable for venting in high vacuums.

Figure 5 shows a vent, 15, connected to the conduit, 2, for directly running off liquid at the wall, 16.

The apparatus according to the present invention may be used as described in the following Examples.

Example 1

For evaporation, a bulk polymer containing from 4.5 to 5 %, by weight, of monomer is sprayed in a quantity of 10 kg/h, together with 4.5 m³/h of nitrogen, into the inner tube through a two-component nozzle at a temperature of 200°C and under a pressure of 0.7 atms gauge and charged with nitrogen in a quantity of 5.5 m³/h through the lateral nozzles of the conduits.

A 100 % pure polymer was obtained at the bottom of the separation vessel, showing that all the monomers had been vapourised and then separated.

Example 2

By mixing and completely reacting 50.1 l/h of oxime with 23.8 l/h of oleum in the presence of 300 Nm³/h of nitrogen using the device of the present invention having a recirculation volume of 1125 l/h, 50.1 l/h of lactam are produced, the reaction temperature not exceeding 100°C by virtue of the measured, finely dispersed addition of the oleum. The cooling water temperature in the tempering jacket was 30°C.

Example 3

100 l/h of highly viscous silicone oil and 120 Nm³/h of nitrogen are passed through a two-phase flow tube. Just upstream of the evaporator exit, 70 % of the gas was run off through a side conduit. In this way, the end of the tube could be positioned in the actual intake of the discharge member at the bottom of the separation vessel, thereby eliminating the problem of product caking on the walls of the separation vessel situated

between the tube and the discharge member. Since a reduced quantity of gas is used, there is little danger of the intake of the discharge member at the bottom of the separation vessel being flushed free.

5 WHAT WE CLAIM IS:

- 10 1. An apparatus for mixing, reacting and/or evaporating components which comprises a helically wound tube, a feed means projecting through the closed inlet end of the tube and a separation vessel at the outlet end of the tube, the coiled tube being surrounded by a tempering jacket divided into sections and one or more enclosed conduits extending laterally from the tube, projecting into the interior of the tube and, when more than one conduit is present, being arranged along the axis of the coiled tube.
- 20 2. An apparatus as claimed in claim 1 in which the feed means is in the form of a two-component nozzle as hereinbefore defined.
- 25 3. An apparatus as claimed in claim 1 or claim 2 in which the end(s) of the conduit(s) located in the coiled tube is (are) in the form of a nozzle(s).
- 30 4. An apparatus as claimed in claim 1 or claim 2 in which the end(s) of the conduit(s) in the coiled tube is (are) in the form of opening(s) for the discharge of fluid.
- 35 5. An apparatus as claimed in any of claims 1 to 3 in which the or each conduit is connected to a metering device.
6. An apparatus as claimed in any of claims 1 2 or 4, in which the or each conduit is connected to an evacuation system.
- 40 7. An apparatus as claimed in any of claims 1 to 6 in which the diameter of the coiled tube decreases towards the tube outlet end.
8. An apparatus as claimed in claim 1 substantially as herein described.
- 45 9. An apparatus as claimed in claim 1 substantially as herein described with reference to any one of the Examples and/or accompanying drawings.

50 ELKINGTON & FIFE,
Chartered Patent Agents,
High Holborn House,
52/54 High Holborn,
London, WC1V 6SH.
Agents for the Applicants.

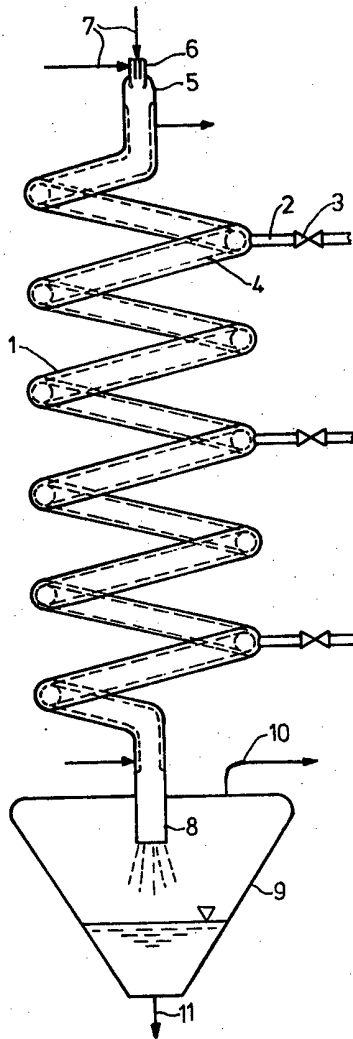
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FIG.1



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FIG. 2

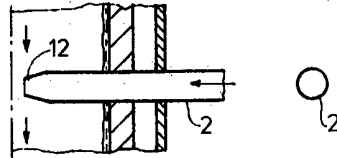


FIG. 3

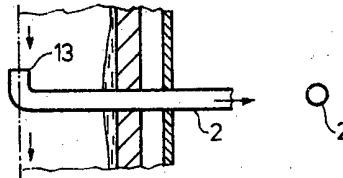


FIG. 4

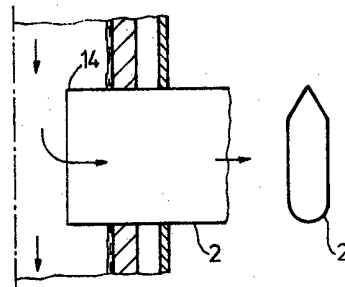


FIG. 5

