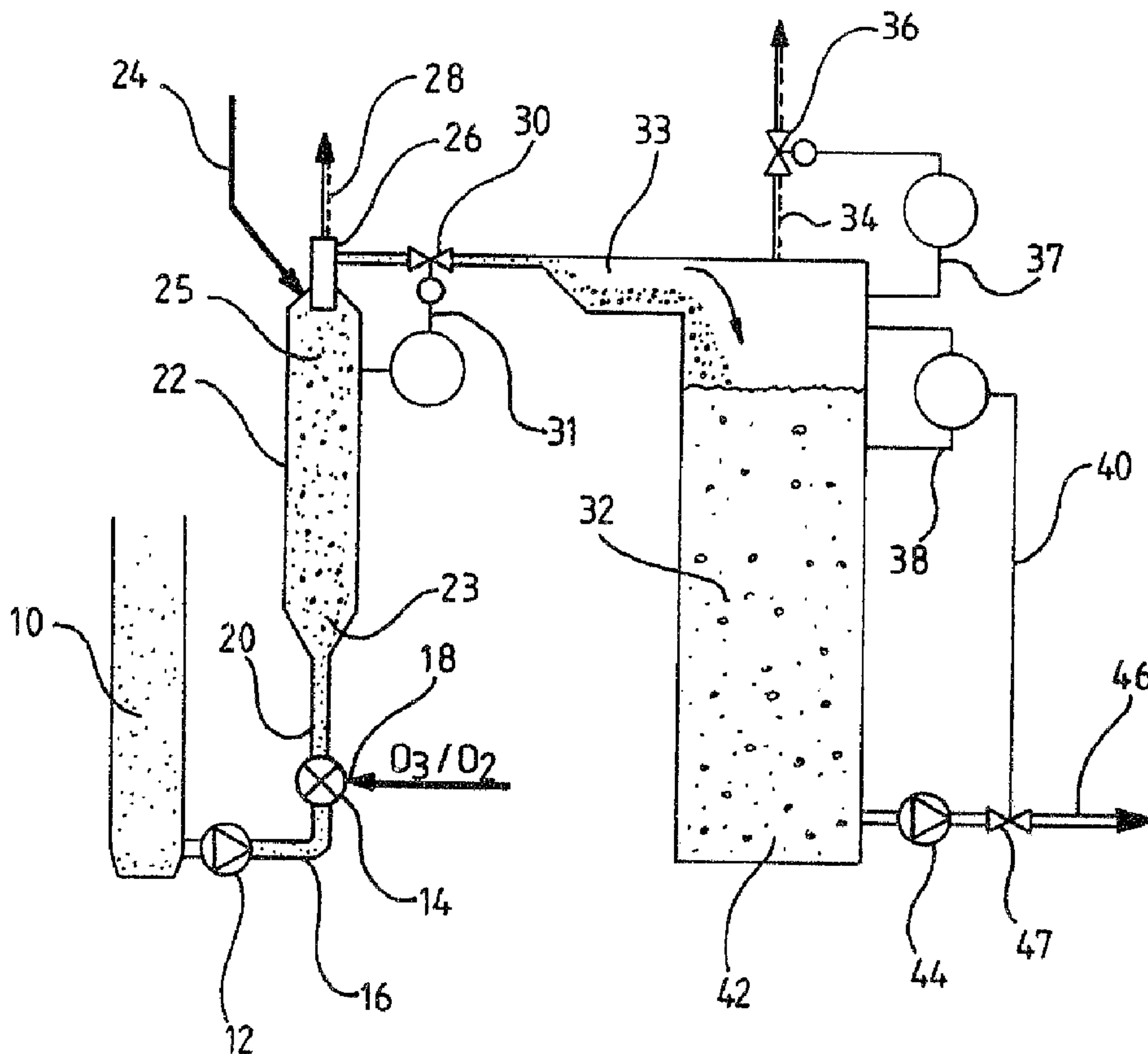




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(54) Titre : BLANCHIMENT A L'OZONE
 (54) Title: OZONE BLEACHING PROCESS



(57) Abrégé/Abstract:

Method and apparatus for ozone bleaching a medium consistency cellulosic fiber suspension include feeding the fiber suspension and an ozone in carrier gas stream under pressure into a fluidizing mixer (14); intimately and uniformly mixing the fiber suspension

(57) **Abrégé(suite)/Abstract(continued):**

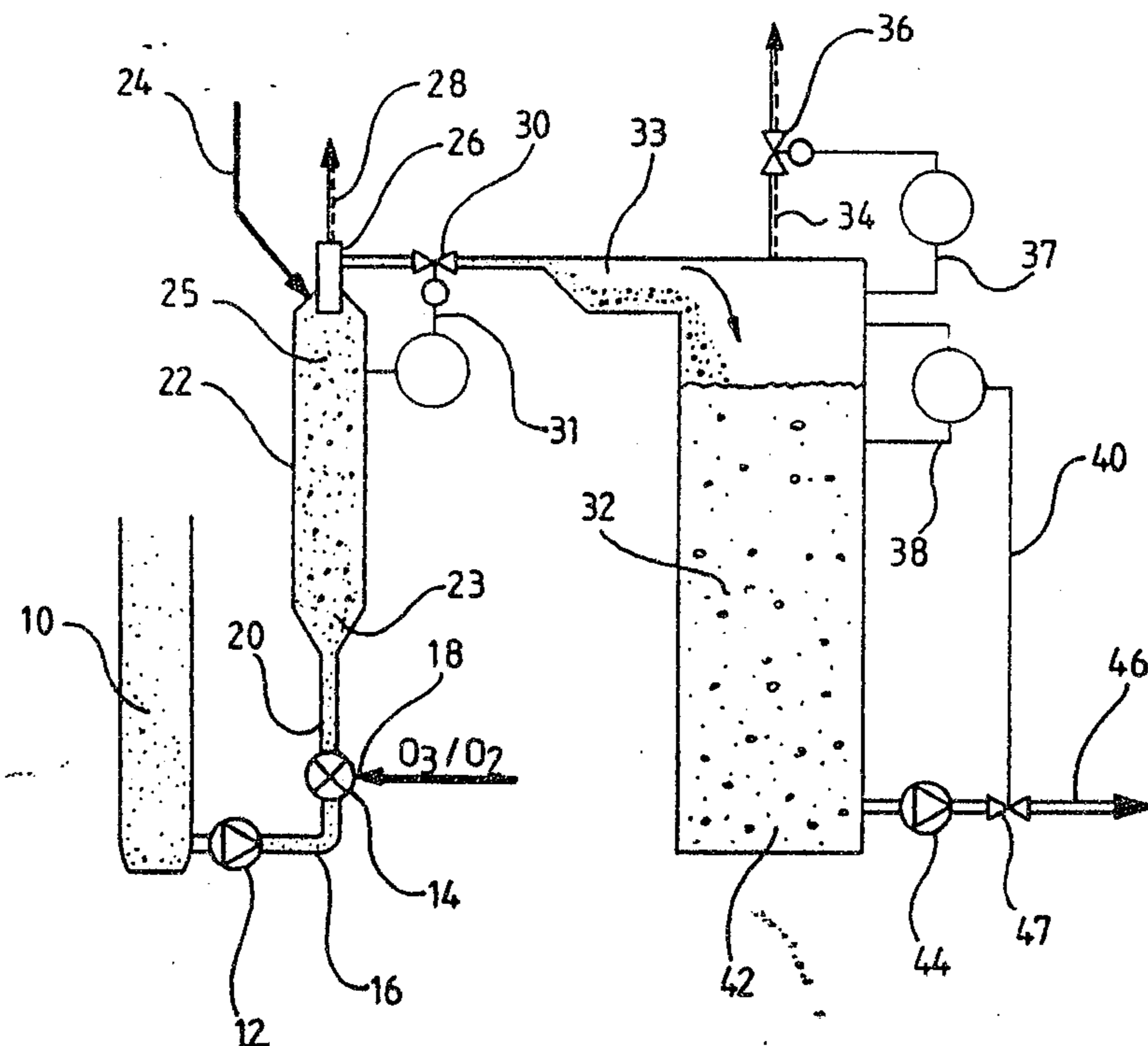
with the ozone to achieve a bleaching reaction; passing the mixture into a first reaction vessel (22) to permit the bleaching reaction to proceed and to consume a major part of the ozone; adding a second bleaching chemical to the mixture; separating the excess ozone and carrier gas from the mixture in a second considerably larger vessel (32) and removing the fiber suspension from the second vessel after the effective second bleaching reaction.



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<p>(21) International Application Number: PCT/FI92/00198 (22) International Filing Date: 25 June 1992 (25.06.92) (30) Priority data: 721,958 27 June 1991 (27.06.91) US (71) Applicant: A. AHLSTROM CORPORATION [FI/FI]; SF-29600 Noormarkku (FI). (72) Inventors: HENRICSON, Kaj ; Eteläpuistokatu 2 B, SF-48100 Kotka (FI). GREENWOOD, Brian ; 8 Van Court, Queensbury, NY 12804 (US). (74) Agent: A. AHLSTROM CORPORATION; Patent Department, P.O. Box 18, SF-48601 Karhula (FI).</p>	<p>(81) Designated States: AU, BR, CA, FI, JP, NO, RU, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE). Published With international search report, With amended claims. <i>ABT. 19</i></p>	

(54) Title: OZONE BLEACHING PROCESS



(57) Abstract

Method and apparatus for ozone bleaching a medium consistency cellulosic fiber suspension include feeding the fiber suspension and an ozone in carrier gas stream under pressure into a fluidizing mixer (14); intimately and uniformly mixing the fiber suspension with the ozone to achieve a bleaching reaction; passing the mixture into a first reaction vessel (22) to permit the bleaching reaction to proceed and to consume a major part of the ozone; adding a second bleaching chemical to the mixture; separating the excess ozone and carrier gas from the mixture in a second considerably larger vessel (32) and removing the fiber suspension from the second vessel after the effective second bleaching reaction.

OZONE BLEACHING PROCESS

Field of the Invention

The present invention relates to ozone bleaching of a medium
5 consistency suspension of cellulosic fibers such as paper pulp
and, particularly, to a method of sequentially bleaching pulp
by ozone and a second bleaching agent, preferably, an alkali
agent to obtain a ZE bleaching sequence.

Background of the invention

10 Bleaching of medium consistency paper pulp with ozone has only
recently become possible and is described in more detail in
U.S. patent number 5,411,633 which issued May 2, 1995
(Phillips et al.). Previous draw backs, e.g. the high cost of
ozone, the known disadvantages associated with operating at
15 either low consistency (less than about 5%) or at very high
consistency (above about 25%) and the fact that ozone readily
attacks the carbohydrates of the pulp, have now been overcome.
Due to the present invention the efficiency of an ozone
bleaching operation of medium consistency pulp, i.e. a
20 cellulosic fiber suspension having a consistency of from about
5 to about 20 percent, is further increased by incorporation
of an additional chemical feeding step into the ozone
bleaching stage.

Bleaching of medium consistency pulp is also known from a
25 European patent application EP-0 426 652 A1. The publication
discloses a method in which medium consistency pulp is
introduced into a so called fluidizing mixer into which also
ozone with carrier gas is introduced. From the mixer the
mixture of ozone and pulp is discharged to a reaction tube
30 from where the pulp is transferred to a bleaching tower via a
pressure decreasing throttling. Between the throttling and
the bleaching tower there has been arranged

a degassing device and a feed conduit for dilution water. In other words, the dilution water is introduced into a pulp the pressure of which has already been decreased to equal with the atmospheric pressure. The specification
5 also suggests that after the above described ozone bleaching stage the pulp may be treated, for instance, by means of an alkaline extraction stage.

There is yet another publication disclosing ozone
10 bleaching, namely US patent specification 4,450,044 which, however, teaches a totally different method of bleaching. The publication relates to a so called high consistency ozone bleaching in which the consistency of the pulp is between 25 and 60 %, preferably between 30 and 45 %.
15 However, the above defined consistencies mean in reality that the pulp is substantially dry, in other words, there is no free water between the fibers. Due to the low, or in this case negligible, water content high consistency ozone bleaching has no problems with regard to water whereas in
20 medium consistency ozone bleaching the biggest problem is the presence of free water between the fibers. Our present invention is directed to solving said problem relating to the presence of free water between the fibers and its effects on the behaviour of the fiber suspension.

25.

Summary of the invention

In accordance with the present invention medium consistency pulp, i.e. a cellulosic fiber suspension having a consistency of from about 5 - 20 % is bleached with ozone
30 by (a) feeding said fiber suspension and an ozone containing carrier gas under pressure preferably at about 3 to about 25 bar, more preferably at about 5 - 14 bar, to a mixer effecting high shear mixing for intimately and homogeneously intermixing the ozone with the medium consistency fiber

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suspension. As a carrier gas oxygen, air and nitrogen may be used, with oxygen being presently preferred as it contains the greatest amount of ozone, namely, about 3 - 16 % at the most. Thus, for example, an ozone carrier gas mixture may contain for instance about 10 kg ozone and 90 kg oxygen. At a pulp suspension consistency of 10 %, the water/gas ratio is preferably between about 1:10 and 2:1 depending on the pressure which varies between 3 to about 25 bar. (b) In the high shear mixer, which is preferably a commercially available MC[®] mixer, the ozone in carrier gas and the paper pulp are thoroughly mixed so that an adequate transfer and contact between the ozone and the fibers is achieved resulting in high bleaching efficiency. (c) From the mixers, the intimate and uniform paper pulp/ozone mixture is passed into a first reaction vessel for allowing the bleaching process to proceed until a major part of the ozone is consumed. The residence time of the mixture in the fluidizing mixer is less than 1 second and the residence time of the mixture of paper pulp and ozone in carrier gas in the first reaction vessel is about 0. 1 to 5. 0 minutes. This permits about 99 % of the ozone to be consumed and the bleaching process to be substantially completed. A second chemical agent, preferably a known bleaching agent such as sodium hydroxide, hydrogen peroxide or chlorine dioxide is added to the mixture in liquid form and also intimately mixed therewith. Preferably, the top of the reaction vessel is provided with a known fluidizing device which fluidizes the contents of the reaction vessel for discharging the mixture into a second vessel for permitting the excess ozone, the carrier gas and a minor amount of possible additional reaction gases to separate from the mixture and also to permit the second bleaching reaction to proceed. To this end, this preferably alkaline mixture of paper pulp may reside in the second vessel, which has preferably a considerably larger cross-section than the first reaction vessel, for up to about 1 - 3 hours. The paper pulp which has now been subjected to a ZE bleaching sequence is then discharged from the bottom of the second

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vessel either to a washer or to another treatment stage including another bleaching stage in a bleaching sequence. Preferably, the pressure in the first reaction vessel is maintained at a predetermined level by a suitable valve and control loop. The pressure in the second vessel is also controlled with known means, albeit at a lower level relative to the pressure in the first vessel. In addition, a suitable known control device is provided to maintain the level of the paper pulp within the second vessel at least within a predetermined range.

Excess gas may be vented at various locations, such as, for example, from the first reaction vessel through the fluidizing device at the top of the reaction vessel. In this connection it should be kept in mind that a constant pressure should be maintained in the first reaction vessel to achieve maximum ozone bleaching effect. Also, the injection under pressure of the liquid bleaching agent is performed at or in close proximity to the fluidizing operation to intimately and uniformly mix the preferably alkaline bleaching agent with the pulp. Finally, both the first and second vessels are preferably upright reactors, whereby the pulp is passed through the first vessel in an upward direction of flow while the pulp passes through the second vessel in a downward flow.

Other objects and features of the present invention will become apparent from the following detailed description considered in conjunction with the accompanying drawings. It is to be understood, however, that the drawings are designed solely for the purposes of illustration and not as a definition of the limits of the invention, for which reference should be made to the appended claims.

35 Brief description of the drawings

The present invention is further described in detail below with reference to the accompanying drawings in which:

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FIG. 1 is a schematic illustration of a first embodiment of the present invention; and
FIG. 2 is a schematic illustration of a second embodiment of the present invention.

5

Detailed description of the presently preferred embodiments

In the figures the same elements are given the same numerals with the exception that all numbers in Fig. 2 are preceded by the numeral 1.

10

Referring now to Fig. 1 in detail, pulp is transferred from a storage unit 10 by a pump 12 which is preferably a degassing medium consistency pump through a line or conduit 16 to a fluidizing mixer 14. Mixer 14 is preferably a high-shear, medium consistency mixer commercially available from Kamyr Inc. of Glens Falls, N.Y. The mixer has an inlet for the medium consistency pulp suspensions connected to line 16, and an inlet port 18, for the pressurized feed of ozone containing carrier gas such as air, nitrogen and preferably oxygen. The pulp suspension is intimately and uniformly mixed with the ozone containing carrier gas and discharged through a mixture outlet into a conduit 20 and passed within about 2 - 3 seconds into the bottom 23 of a first upright reaction vessel 22. After a residence time of about 0.5 to 5 min. the mixture of pulp, carrier gas and ozone, which has not yet been entirely consumed during the bleaching reaction, arrives at the top 25 of the reaction vessel 22. Into the reaction mixture is now fed through a conduit or line 24, at the top 25 of the reaction vessel 22 an additional bleaching chemical, for example, sodium hydroxide, hydrogen peroxide, sodium peroxide, chlorine dioxide, or the like, preferably in liquid form and if necessary with a carrier, under pressure. Preferably, the bleaching chemical is intimately mixed with the paper pulp to effect the alkaline bleaching step.

35

To assist the removal of the paper pulp from the first reaction vessel, vessel 22 is provided at the top portion

25 thereof with a known fluidizing device or fluidizing
discharger 26 which preferably has an integral injection
port for the bleaching chemical at or near the fluidizing
rotor or fluidizing device or discharger 26 so as to effect
5 the proper mixing of the bleaching chemical with the
fluidized paper pulp. The fluidizing discharger 26 is
preferably provided with means 28 for discharging
preferably pressurized gas from the reactor to be used,
for example, in another pressurized bleaching stage. To
10 achieve good bleaching results and stable conditions the
pressure in the first reaction vessel 22 should be
maintained at a constant level which is achieved with a
pressure regulating valve 30, preferably located closely
adjacent fluidizing discharger 26, and control loop 31 in
15 known manner.

The bleached pulp, which now contains excess ozone, carrier
gas and bleaching chemical, for example, sodium hydroxide,
is now discharged from first vessel 22, preferably into
20 the enlarged inlet portion 33 of a second upright vessel
32 to assist in the separation of the gasses from the pulp
mixture. Separated gas is then removed from the second
vessel 32 through a gas discharge line 34. The pressure
in the second vessel 32 is also maintained steady, albeit
25 at a relatively substantially lower level than in the
first vessel, generally only at slight overpressure, by a
separate pressure regulating valve 36 and a control loop
37 in a known manner. The pulp is now collected in the
second vessel 32 at or near a predetermined level, through
30 known level control means 38, line 40, pump 44, and pressure
regulating valve 47 for up to 1 to 3 hours to complete the
alkaline (E) bleaching stage and thereafter is discharged
at the bottom 42 of vessel 32 by a pump 44, which is
preferably also a degassing medium consistency pump, through
35 a valve 47 into a conduit 46 leading to a washer or other
suitable treatment stage.

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The elements, structure and operation of the embodiment illustrated in Fig. 2 are substantially the same as the embodiment described above in connection with Fig. 1, except that second vessel 132 is provided at its bottom 5 142 with an outlet 152 which is dimensioned to permit the bleached paper pulp to be fed, due to the pressure head thereof, into a suitable washer 150, preferably a drum diffusion washer as sold by assignee A. Ahlstrom Corporation, with pressurized inlet or diffuser available 10 from Kamyr Inc. of Glens Falls, N.Y.

A level control mechanism 138 cooperates through line 148 with an rpm regulator of washer 150 in known manner to maintain the paper pulp level in the second vessel 132 at 15 a predetermined level. Finally, the washed pulp is discharged from washer 150 by a pump 144, preferably a degassing medium consistency pump through a conduit 146 for further treatment.

20 It is understood that additions and modifications can be made to the described embodiments which are within the scope of the present invention. The description is thus not to be construed as limiting but only as exemplary, the scope of the invention being properly delineated only 25 in the appended claim.

Thus, while there have been shown and described and pointed out fundamental novel features of the invention as applied to preferred embodiments thereof, it will be understood 30 that various omissions and substitutions and changes in the form and details of the disclosed invention may be made by those skilled in the art without departing from the spirit of the invention. It is the intention, however, therefore, to be limited only as indicated by the scope of 35 the claims appended hereto.

CLAIMS:

1. A method of bleaching cellulose pulp of a consistency of 5 to 20 percent with ozone as bleaching agent, utilizing a fluidizing mixer, and first and second reaction vessels, the second vessel larger than the first, comprising the steps of continuously:
 - (a) feeding the pulp and ozone gas in substantially non-consumable carrier gas under a pressure of 3 to 25 bar into the fluidizing mixer;
 - (b) intimately mixing the pulp and ozone in the fluidizing mixer to produce a uniform and intimate mixture of pulp and ozone;
 - (c) while maintaining the mixture under a pressure of 3 to 25 bar, feeding the mixture to the first reaction vessel;
 - (d) retaining the mixture in the first reaction vessel while it moves in a first direction until bleaching with the ozone in the mixture has been substantially completed;
 - (e) after substantial completion of the ozone bleaching reaction, while a pressure of 3 to 25 bar is maintained, introducing a second bleaching chemical, different from ozone, into the pulp in the first reaction vessel;
 - (f) fluidizing the pulp, with second bleaching chemical, in the first reaction vessel; and
 - (g) discharging the fluidized pulp in a second direction different than the first direction into the second reaction vessel, gas, including ozone carrier gas, separating from the pulp in the second reaction vessel; and
 - (h) discharging pulp from the second reaction vessel after the second bleaching chemical has substantially completely reacted with the pulp.

2. A method as recited in claim 1 wherein the pressure in the second vessel is substantially less than in the first vessel.
3. A method as recited in claim 1 wherein steps (a), (c) and (e) are practiced with the pulp maintained at a pressure of about 5-14 bar.
4. A method as recited in claim 1 wherein step (e) is practiced by introducing a liquid bleaching agent as the second bleaching chemical.
5. A method as recited in claim 4 wherein step (e) is practiced using as the second bleaching chemical a liquid agent selected from the group consisting essentially of sodium hydroxide, hydrogen peroxide, and chlorine dioxide.
6. A method as recited in claim 1 comprising the further step of removing the gas that separates from the pulp from the top of the second reaction vessel.
7. A method as recited in claim 1 wherein step (c) is practiced by passing the mixture substantially upwardly, the first direction being substantially vertically upward.
8. A method as recited in claim 7 comprising the further step of passing the pulp substantially vertically downwardly in the second reaction vessel.
9. A method as recited in claim 1 comprising the further step of washing the pulp in the second vessel after reaction with the second bleaching chemical in the second reaction vessel takes place.
10. A method as recited in claim 1 wherein step (e) is practiced using sodium hydroxide as the second bleaching chemical.

11. A method of bleaching cellulose pulp of a consistency of 5 to 20 percent with ozone as bleaching agent, utilizing a fluidizing mixer, and first and second reaction vessels, the second vessel larger than the first, consisting essentially of the steps of:
- (a) feeding the pulp and ozone gas in substantially non-consumable carrier gas under a pressure of 3 to 25 bar into the fluidizing mixer;
 - (b) intimately mixing the pulp and ozone in the fluidizing mixer to produce a uniform and intimate mixture of pulp and ozone;
 - (c) while maintaining the mixture under a pressure of 3 to 25 bar, feeding the mixture to the first reaction vessel;
 - (d) retaining the mixture in the first reaction vessel while it moves in a first direction until bleaching with the ozone in the mixture has been substantially completed;
 - (e) after substantial completion of the ozone bleaching reaction, while a pressure of 3 to 25 bar is maintained, introducing a second bleaching chemical, different from ozone, into the pulp in the first reaction vessel;
 - (f) fluidizing the pulp, with second bleaching chemical, in the first reaction vessel;
 - (g) discharging the fluidized pulp in a second direction different than the first direction into the second reaction vessel, gas, including ozone carrier gas, separating from the pulp in the second reaction vessel; and
 - (h) discharging pulp from the second reaction vessel after the second bleaching chemical has substantially completely reacted with the pulp.

12. A method as recited in claim 11 wherein the pressure in the second vessel is substantially less than in the first vessel.
13. A method as recited in claim 11 wherein step (e) is practiced by introducing a liquid bleaching agent as the second bleaching chemical.
14. A method as recited in claim 11 comprising the further step of removing the gas that separates from the pulp from the top of the second reaction vessel.
15. A method as recited in claim 11 wherein step (c) is practiced by passing the mixture substantially upwardly, the first direction being substantially vertically upward, and wherein the pulp moves downwardly in the second vessel.
16. An apparatus for ozone bleaching a fiber suspension of a consistency of 5 to 20 percent, said apparatus comprising a fluidizing mixer comprising a pulp inlet, an ozone in carrier gas inlet and an outlet for a mixture containing pulp, ozone and carrier gas;
- a pressurized first upflow reaction vessel having a bottom portion connected to said mixture outlet of said fluidizing mixer and a top portion;
- a pressure regulating valve operatively connected to said top portion of said first reaction vessel; and
- a second down flow vessel comprising a top portion, an inlet at said top portion connected to said top portion of said first reaction vessel via said pressure regulating valve, and a bottom portion; and
- means connected to said bottom portion of said second vessel for removing said fiber suspension therefrom;

characterized in means for feeding a bleaching chemical to said fiber suspension at a locus between and including said top portion of said first reaction vessel and said pressure regulating valve.

17. The apparatus of claim 16, characterized in means at said top portion of said first reaction vessel for fluidizing said mixture to assist the feeding of said mixture through said pressure regulating valve into said inlet of said second vessel.

18. The apparatus of claim 17, characterized in that said means for feeding said chemical feeds said chemical into said first vessel and is a part of said fluidizing means.

19. The apparatus of claim 16, wherein;

the gas inlet operatively connected to said fluidizing mixer feeds said ozone in carrier gas under pressure to said mixer.

20. The apparatus of claim 16, characterized in that said first and second vessels are pressurized.

21. The apparatus of claim 20, characterized in that said pressure in said first vessel is between about 3 and 25 bar.

22. The apparatus of claim 16, characterized in that said chemical feeding means comprises means for feeding a liquid chemical to said first vessel.

23. The apparatus of claim 16, characterized by means operatively connected to said first reaction vessel for separating and removing gas from said first reaction vessel.

24. The apparatus of claim 16, characterized by means operatively connected to said second vessel for separating and removing gas therefrom.

25. The apparatus of claim 20 or 21, characterized in that said pressure in said first vessel is between about 5 to 14 bar.

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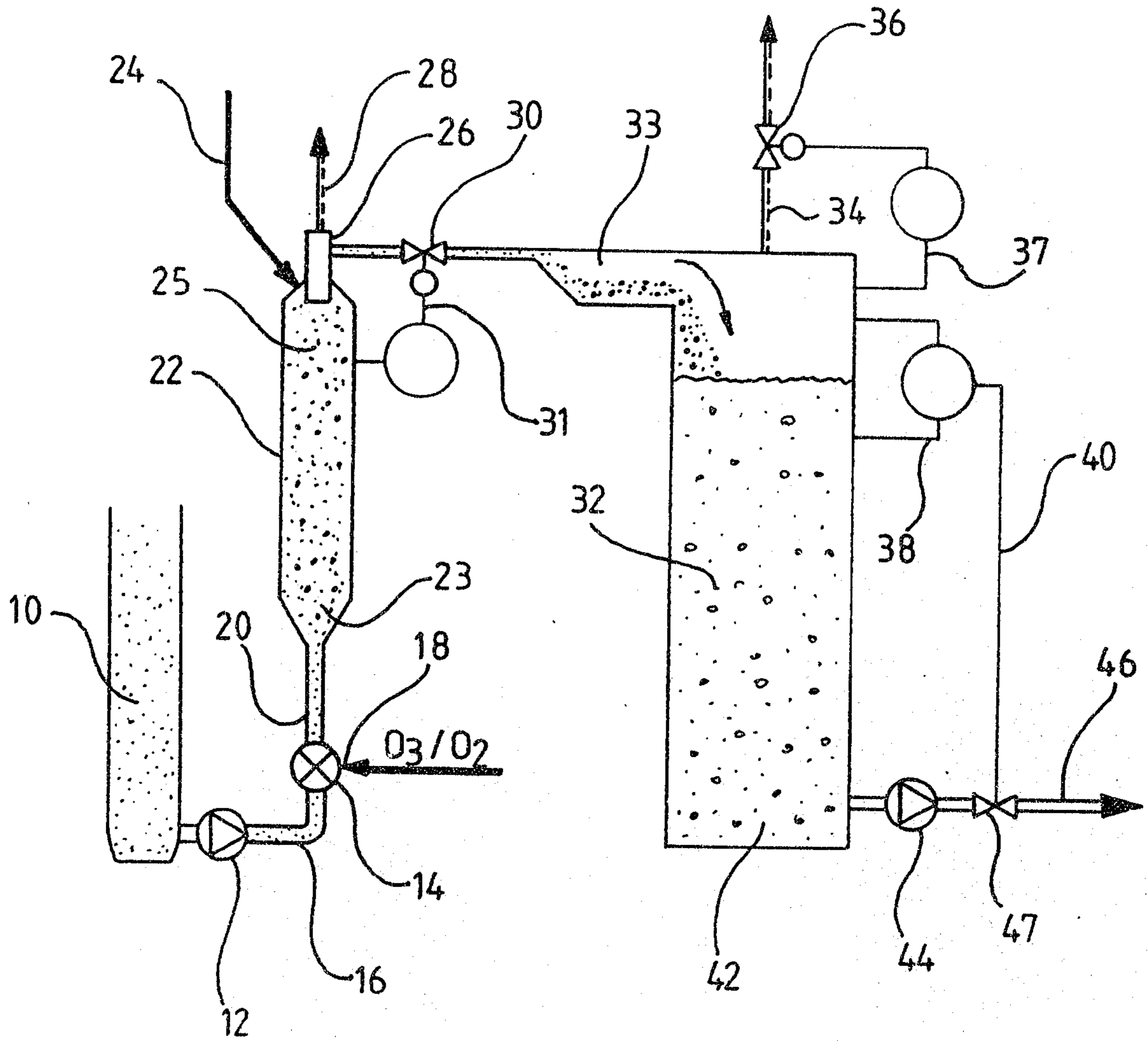


FIG. 1

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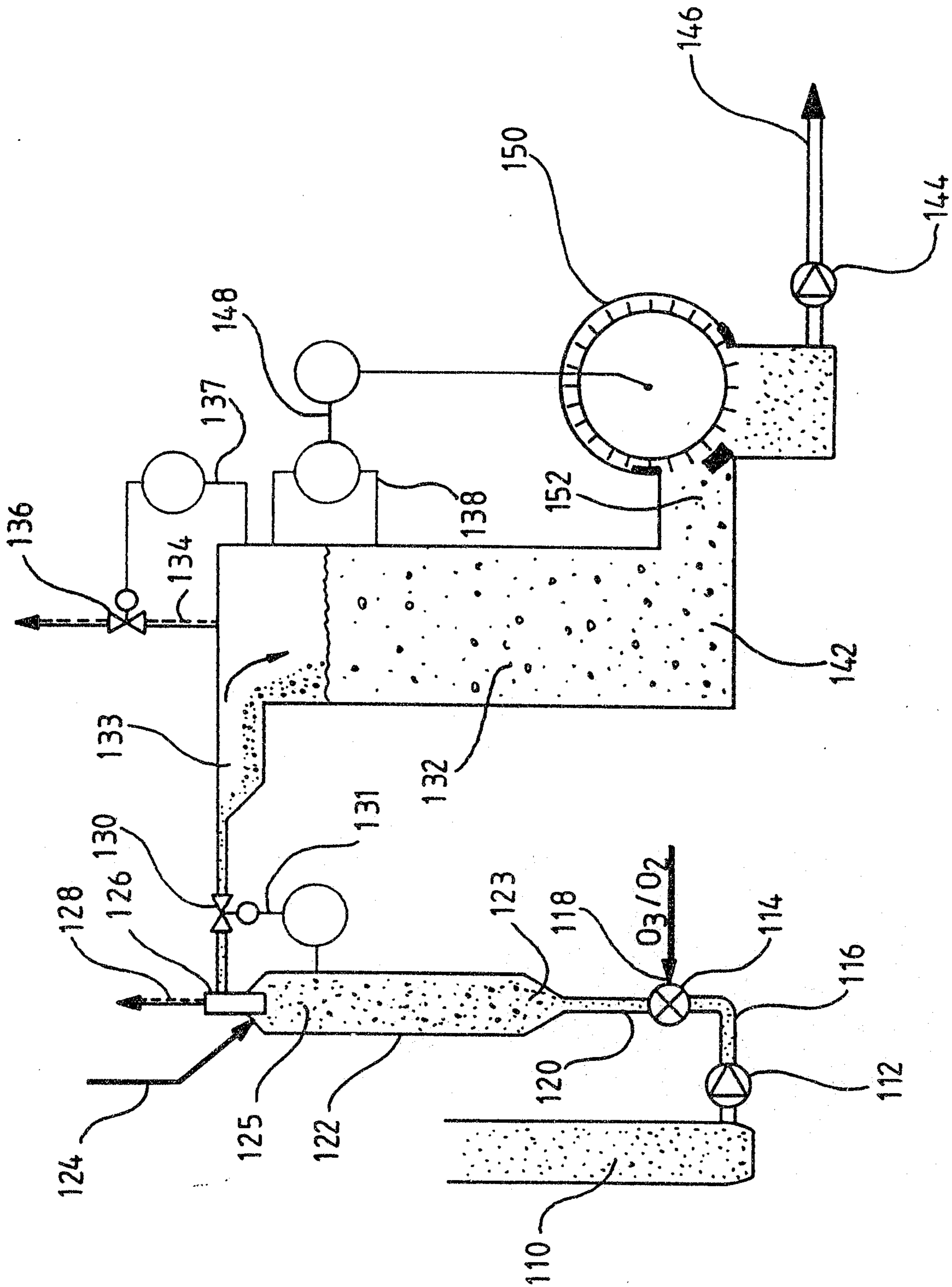


FIG. 2

