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(54) Pneumatic reactor ozone bleaching of paper pulp.

(57) A method and apparatus (10) for delignifying comminuted cellulosic fibrous material (paper pulp) with ozone. Pulp having a consistency of about 25-45% is fluffed (in 16), ozone is added to it (17, 19), and then it is pneumatically conveyed (by 20) in a pathway (21) for at least several seconds (e.g. about four seconds). The majority of the gas is then separated from the pulp in a cyclone (22), and the pulp is discharged into a retention tube (25), where it is retained for at least a few seconds (e.g. about ten seconds) in a relatively static condition. It is then fed through an air lock (26) to another blower (120), and additional ozone added to it (via 35), then the above steps are repeated. The gas separated from one cyclone (123, 223) can be returned as conveying gas to another blower (20, 120). The method is practiced at a pH of about 1.8-3.5, and a temperature of about 20 to 50 ° C.

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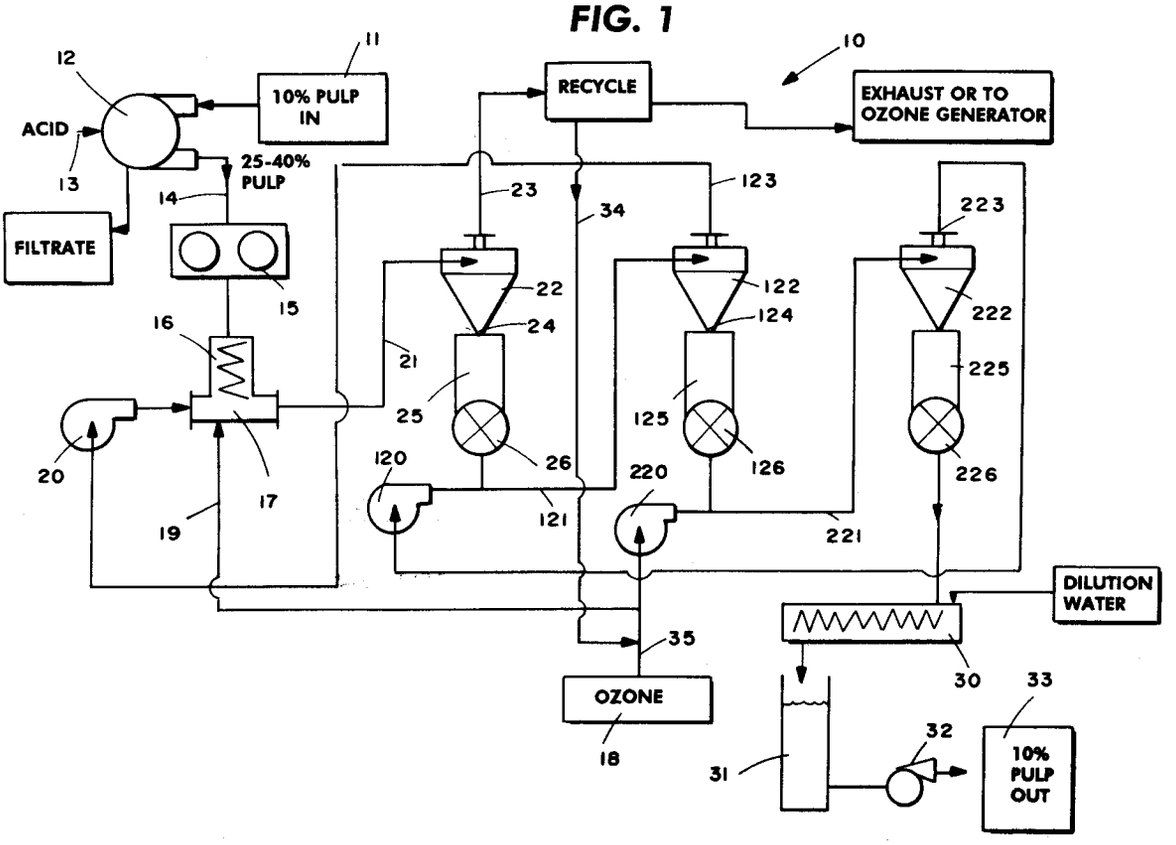
FIG. 1


10% PULP IN
RECYCLE
EXHAUST OR TO OZONE GENERATOR
ACID
25-40% PULP
FILTRATE
DILUTION WATER
OZONE
10% PULP OUT
10
11
12
13
14
15
16
17
18
19
20
21
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226

BACKGROUND AND SUMMARY OF THE INVENTION

The desirability of bleaching (delignifying) comminuted cellulosic fibrous material (paper pulp) with ozone has been known for more than 100 years. However despite known advantages for delignifying pulp with ozone, as of the present time there are no known significant commercial installations for effecting ozone delignification. This is due in part to the difficulties in controlling the delignifying reaction, which is extraordinarily rapid. If the pulp is held in contact with ozone too long -- for example if the pulp forms into wads or slugs so that only a small percentage of the surface area thereof is actually contacted with ozone gas -- the ozone will not only effect delignification, but will start to degrade the cellulose, producing pulp having poor strength properties. This is so even if viscosity protectors are utilized. Prior proposals for ozone reactors include at least some mechanical elements which contact the pulp, and which have the tendency to form the pulp into clumps.

According to the present invention, a method and apparatus are provided for reacting pulp with ozone in such a way that the maximum amount of surface area of the pulp will be exposed to the ozone at all times. Also, after a relatively short period of time of having the pulp in contact with ozone, most of the ozone containing gas is removed and the pulp is retained generally static for several seconds, at which time the delignification proceeds to completion, or the pulp is subjected to another exposure (or multiple exposures) to ozone containing gas. According to the invention, no mechanical elements are utilized to effect exposure of the pulp to ozone containing gas, but rather the primary reactions take place as the pulp is being pneumatically conveyed.

According to one aspect of the present invention, a method for delignifying comminuted cellulosic fibrous material with ozone containing gas is provided. The method comprises the steps of continuously and sequentially: (a) Fluffing the material. (b) Adding a gas, containing an effective amount of ozone to effect delignification, to the material. (c) Pneumatically conveying the material in a first path for at least a few seconds, the ozone reacting with the material during conveyance to effect delignification. (d) Separating the majority of gas from the material at the end of the first path. And, (e) maintaining the material in a generally static state for at least a few seconds. The material typically has a consistency of about 25-45% during treatment, a pH of about 1.5-4.0, and is at a temperature of about 20-50 ° C. Normally the step (f) of repeating steps (b)-(e), at least one time, is also practiced. Desirably the gas providing pneumatic conveyance contains ozone recycled from a subsequent separating step, and the primary supply gas is oxygen so that the amount of ozone in the supply gas can be maximized (e.g. about 4-8% by weight with current technology for ozone production, so that the application on pulp is up to 1.5% and preferably one-half percent by weight). Step (c) is preferably practiced for about four seconds, step (d) for about one second, and step (e) for about ten seconds.

According to another aspect of the present invention, apparatus for effecting ozone delignification of pulp is provided. The apparatus comprises: A fluffer having an inlet and a discharge. Means for adding ozone containing gas to the discharge from the fluffer. Gas moving means connected to the discharge for the fluffer for entraining material mixed with ozone in a gas stream and conveying it. A conduit connected to the gas moving means for conveying material with ozone gas therethrough. A gas/material separator connected to the conduit remote from the gas moving means, the separator having a gas discharge and a material discharge. A retention tube connected to the material discharge from the separator, and having a discharge therefrom. And, an air lock connected to the discharge from the retention tube. The gas/material separator is preferably a cyclone, while the gas moving means is a blower, and gas from a downstream cyclone is provided as feed gas to an upstream blower.

It is the primary object of the present invention to provide for the effective delignification of pulp using ozone, while minimizing the possibility that the ozone will attack the cellulose and degrade pulp strength. This and other objects of the invention will become clear from an inspection of the detailed description of the invention, and from the appended claims.

BRIEF DESCRIPTION OF THE DRAWING

FIGURE 1 is a schematic diagram illustrating exemplary apparatus according to the present invention useful in ozone delignification of pulp.

DETAILED DESCRIPTION OF THE DRAWINGS

An exemplary apparatus 10 according to the present invention, for practicing the method of ozone delignification of pulp according to the present invention, is illustrated schematically in FIGURE 1. A source of pulp (e.g., at about 1-12% consistency) from source 11 is thickened, as in a press 12, and acid 13 is added thereto so as to bring its pH in the range of about 1.5 to 4.0, desirably about 1.8-3.5, and preferably about 2.5-3.0. The pulp discharged in line 14 from the press

12 has a higher consistency, typically over 20%, and preferably about 25-45%. At that consistency, it typically is in almost the form of a log or a sheet depending on the type of press, with many of the fibers held together in clumps. It is fed to a conventional shredder 15 which breaks up these large clumps into much smaller clumps, and then is fed to a conventional fluffer 16 which fluffs the pulp into very small particles, preferably individual fibers, which will have a very large surface area for exposure to delignifying gas.

At the discharge 17 from the fluffer 16, an ozone carrying gas from source 18 is fed by a conduit 19 to be introduced into and mixed with approximately 25-40% fluffed comminuted cellulosic fibrous material (paper pulp) at approximately 25-45% consistency in the discharge 17. The ozone gas from source 18 typically is provided by an ozone generator, and is in a carrying gas. While air or other gases could be utilized as the carrying gas, since the highest concentration of ozone presently possible in air is only about two to three percent, it is desirable that oxygen gas be used as the carrying gas. In oxygen gas the present technologically practiced maximum content of ozone is about 11 to 12%, while practically about 3 to 8% ozone by weight can be provided. Therefore it is preferred, but not essential, that the gas in conduit 19 comprise about 90-97% O_2, and about 3-8% (or higher if techniques are ever developed to provide a higher percentage of ozone in oxygen) O_3. Of course there will be minor amounts of other gases, such as the gases that make up air, which should have no significant adverse affect on the delignifying action.

In the fluffer discharge 17 the pulp is entrained in a stream of gas exiting from the gas moving means (blower) 20, and the mixture of ozone containing gas and pulp is thereby pneumatically conveyed in a first path, defined by conduit 21. The length of the path defined by conduit 21, and the speed of pneumatic conveyance, are gauged so that the pulp is exposed to the ozone containing gas for at least a few seconds, typically about 15 seconds or less, e.g. about four seconds. Since the pulp in the conduit 21 is fluffy, having a great surface area, and in view of the fact that it is intimately surrounded by the ozone containing gas, being thoroughly mixed therewith without any mechanical elements during the conveyance, the delignifying reaction will be quick and effective.

At the end of the path defined by conduit 21, a gas/pulp separator -- such as the cyclone 22 -- is provided, the cyclone 22 having a gas discharge 23 and a pulp discharge 24. The cyclone 22 allows the charge of ozone to be "slipped", that is it ensures that ozone is not exposed in relatively high concentration to the pulp for such a long period of time that some of the pulp cellulose might be attacked by the ozone. The vast majority of the spent gas -- which includes oxygen and unreacted ozone as the primary constituents -- is discharged in conduit 23 for exhaust or recycle, while the comminuted cellulosic fibrous material -- which has little -- though some -- gas remaining therein is discharged at 24. A portion of the recycled gas is reused as carrier gas through line 34 into line 35. The remaining portion of recycle gas (nearly equal to supply gas) is recycled to an ozone generation, or if not economical for ozone generation it is exhausted.

The discharge 24 from cyclone 22 is preferably into a vessel, such as a substantially vertical axis retention tube 25. In the retention tube 25, the fluffed pulp is maintained in a generally static condition (although there is movement downwardly in the tube) for at least a few seconds, e.g. for about ten seconds (but typically less than a minute), so that a delignifying reaction with a small amount of residual ozone has an opportunity to move toward completion. Then the pulp is discharged by air lock 26 into another sequence of equipment substantially identical to that just described. The air lock 26 preferably is a conventional low pressure feeder, such as used commonly in the pulp and paper art for feeding wood chips to a low pressure steaming vessel.

The second set of equipment to which the air lock 26 is connected is illustrated in FIGURE 1 by the same reference numerals as the first set of equipment only preceded by a "1". That is a blower 120 provides a stream of conveying gas which entrains the fluffed pulp from air lock therein. The gas discharged by the blower 120 has ozone therein, as will be hereinafter described. The path defined by the conduit 121 terminates in a second cyclone 122 which has a gas discharge 123 and a material discharge 124. The gas discharge 123 is connected up to the blower 20 to provide recycle of the gas in the system, the gas in conduit 123 containing some ozone and primarily oxygen. The material is held in retention tube 125 for at least a few seconds, and then discharged into air lock 125.

Any number of sequences of equipment as illustrated in FIGURE 1 can be provided. For example a third set of equipment may be provided, which is illustrated in FIGURE 1 by the same reference numerals as for the first set of equipment only preceded by a "2". That is the fluffed material is discharged from air lock 126 into conduit 221 and pneumatically conveyed therein by the blower 220. Ozone containing gas (typically oxygen) from source 18 is provided to the blower 220 to provide a portion of the gas needed to pneumatically convey the fluffed pulp to the third separator 222. The majority of the conveying gas is spent gas from

line 21 which is passed through line 34 to line 35. Fresh ozone/oxygen mixture is added to line 35. The gas from gas discharge 223 to the third separator 222 is fed back as the feed gas to the blower 120 and includes ozone therein, while the material is discharged from cyclone 222 to retention tube 225 and then into air lock 226.

It will be seen with respect to the apparatus 10 that it is preferred to use recycle gas for all of the gas moving means, to which ozone of about 4-8% (or higher if ever practically reachable) by weight in a supply gas, such as oxygen, is provided. It is desirable to provide up to 1.5% but preferably about one-half percent ozone by weight on the comminuted cellulosic fibrous material to optimally effect delignification.

In the preferred embodiment illustrated in FIGURE 1, the pulp discharged from air lock 226 is fed to a conventional repulper 30, from there into a standpipe 31, from which case it is pumped by a low or medium consistency pump 32 to pulp outlet 33. The discharge consistency can be set at any level required to suit the following washing stage.

While the temperature is not particularly critical in the ozone bleaching process according to the invention, and can vary all the way from less than 0 ° C to more than 80 ° C, preferably the temperature is kept within the range of about 20-50 ° C. Of course, a number of conventional other steps may also be practiced in the method according to the invention, such as adding a conventional protector to the pulp prior to bleaching. Also, other bleaching chemicals, such as hydrogen peroxide, may be utilized in the reactor. Also, multiple addition points of the ozone containing gas may be provided and the gas can be introduced co-currently, or countercurrently. While three sequences of blower, conduit, separator, retention vessel, and air lock have been illustrated, under some circumstances only one sequence need be utilized, or many more can be utilized, depending on the material, the degree of delignification desired, and the effective amount of ozone provided in the conveying gas.

It will thus be seen that according to the present invention a method and apparatus have been provided for effectively intimately mixing pulp with ozone containing gas to facilitate delignification of the pulp, while guarding against the ozone reaction going so far as to attack the pulp cellulose. While the invention has been herein shown and described in what is presently conceived to be the most practical and preferred embodiment thereof, it will be apparent to those of ordinary skill in the art that many modifications may be made thereof within the scope of the invention, which scope is to be accorded the broadest interpretation of the appended so as to encompass all equivalent structures and methods.

Claims

1. A method of delignifying comminuted cellulosic fibrous material, comprising the steps of substantially continuously and sequentially: (a) fluffing the material (in 16); and (b) adding a gas (in 17) containing an effective amount of ozone to effect delignification, to the material; characterized by the steps of:
   (c) pneumatically conveying the material (in 21, with 20) in a first path for at least a few seconds, the ozone reacting with the material during conveyance to effect delignification;
   (d) separating (in 22) the majority of gas from the material at the end of said first path; and
   (e) maintaining (in 25) the material, with a small amount of residual ozone, in a generally static state for at least a few seconds.

2. A method as recited in claim 1 further characterized in that steps (a)-(e) are practiced with the material having a consistency of above about 20% during the practice of steps (a)-(e).

3. A method as recited in claim 2 characterized by the further step (f) of repeating steps (b)-(e) immediately after step (e), at least one time (in 121, 122, etc.).

4. A method as recited in claim 3 further characterized in that steps (a) through (f) are practiced with the material having a consistency of about 25-45%, and a pH of about 1.5-4.0, and the amount of ozone contacting the material is about one-half - 1 1/2 percent by weight.

5. A method as recited in claim 3 further characterized in that the gas separated in step (d) in at least one sequence of steps (123, 223) is provided as ozone containing gas in step (b) in an earlier sequence of steps (20, 120).

6. A method as recited in claim 1 further characterized in that step (c) is practiced so that it is about 15 seconds or less in duration, and step (d) is practiced by cyclone separation (in 22).

7. A method as recited in claim 1 further characterized in that step (c) is about four seconds in duration, and step (e) is about ten seconds in duration.

8. A method as recited in claim 3 characterized by the further step (g) of passing the material through an air lock (26, 126, 226) between step

(e) of one sequence, and step (b) of the next.

9. An apparatus (10) for delignifying pulp comprising:

a fluffer (16) having an inlet and a discharge (17); means (19) for adding ozone containing gas to the discharge from said fluffer; gas moving means (20) connected to the discharge for said fluffer for entraining material mixed with ozone in a gas stream and conveying it; and a conduit connected (21) to said gas moving means for conveying material with ozone gas therethrough; characterized by:

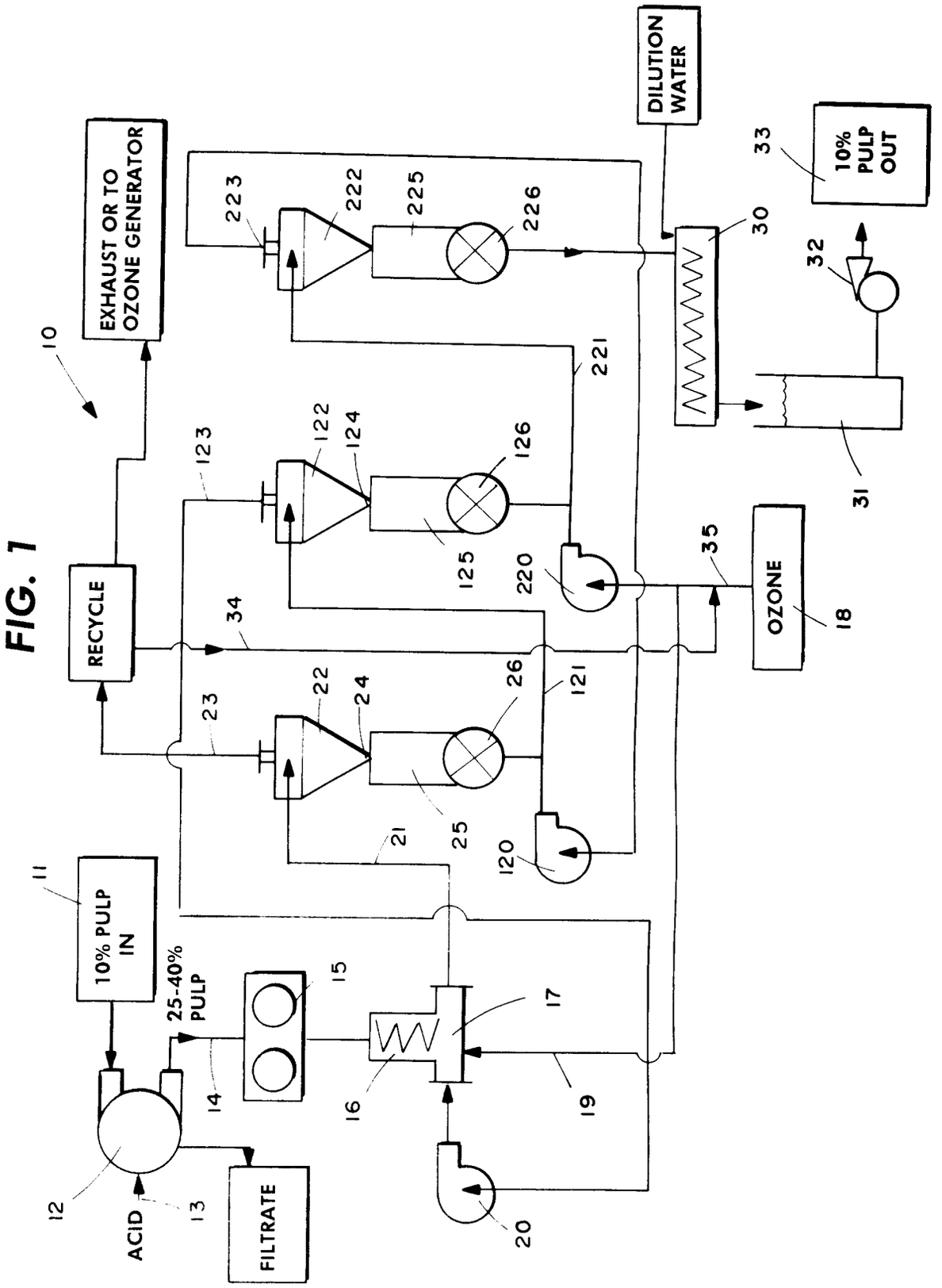
a gas/material separator (22) connected to said conduit remote from said gas moving means, said separator having a gas discharge (23) and a material discharge (24);

a retention tube (25) connected to the material discharge from said separator, and having a discharge therefrom; and

an air lock (26) connected to the discharge from said retention tube.

10. Apparatus as recited in claim 9 further characterized by a second means (35) for adding ozone containing gas to material discharged from said air lock, a second gas moving means (120), a second conduit (121), a second separator (122), a second retention tube (125), a second air lock (126), and a recycle conduit (123) connecting the gas discharged from said second separator to said first blower.

FIG. 1


EXHAUST OR TO OZONE GENERATOR
DILUTION WATER
10% PULP OUT
10% PULP IN
25-40% PULP
RECYCLE
OZONE
FILTRATE
ACID
10
11
12
13
14
15
16
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European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 10 9775

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
P,A	DE-A-4 025 616 (MASCHINENFABRIK ANDRITZ AG) * the whole document *	1-10	D21C9/153
A	EP-A-0 308 314 (DEGREMONT) * the whole document *		
A	FR-A-2 388 933 (MYRENS VERKSTED A/S)		

TECHNICAL FIELDS SEARCHED (Int. Cl.5)
D21C
D21B

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	31 MARCH 1992	SONGY Odile

CATEGORY OF CITED DOCUMENTS

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