

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 1 287 201 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
11.02.2004 Bulletin 2004/07

(51) Int Cl.7: **D21H 21/02**, D21H 21/04,
D21F 1/66

(21) Application number: **01931742.9**

(86) International application number:
PCT/FI2001/000410

(22) Date of filing: **27.04.2001**

(87) International publication number:
WO 2001/083886 (08.11.2001 Gazette 2001/45)

(54) **A PROCESS FOR CONTROLLING MICROBIAL GROWTH**

VERFAHREN ZUR KONTROLLE VON MIKROBIELEM WACHSTUM

PROCESSUS PERMETTANT DE MAITRISER LA CROISSANCE MICROBIENNE

(84) Designated Contracting States:
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE TR**

(30) Priority: **04.05.2000 FI 20001040**

(43) Date of publication of application:
05.03.2003 Bulletin 2003/10

(73) Proprietor: **AGA AKTIEBOLAG**
181 81 Lidingö (SE)

(72) Inventors:
• **HOLMBERG, Anna**
S-426 74 Västra Frölunda (SE)
• **LEINO, Hannu**
FIN-02320 Espoo (FI)
• **KONTOLA, Pia**
FIN-02940 Espoo (FI)
• **PUUTONEN, Pirjo**
A-2380 Perchtoldsdorf (AT)

(74) Representative: **Kasseckert, Rainer et al**
Linde AG
Zentrale Patentabteilung
Dr.-Carl-von-Linde-Strasse 6-14
82049 Höllriegelskreuth (DE)

(56) References cited:
EP-A- 1 002 899 WO-A-88/04706
US-A- 3 806 404 US-A- 4 532 007
US-A- 5 139 613

- **DATABASE WPI Section Ch, Week 198911**
Derwent Publications Ltd., London, GB; Class
F09, AN 1989-078753 XP002901861 -& EP 0 307
295 A (CANADIAN LIQUID AIR LTD AIR LIQUIDE
CANADA LTEE) 15 March 1989 (1989-03-15)
- **DATABASE PAPERCHEM. [Online] Dialog**
information serv., File 240, KYMRON B J: "Safe
nontoxic pest control for books" retrieved from
DIALOG INF. SERV., accession no. 00408118
Database accession no. GA3701113
XP002901862 & THE ABBEY NEWSLETTER, vol.
14, no. 1, 16 February 1990 (1990-02-16),

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 1 287 201 B1

Description

[0001] The present invention relates to a process for controlling microbial growth in a production line for cellulosic products with the aid of gases. The invention also relates to the use of gases such as carbon dioxide, nitrogen, a noble gas and/or non-naturally occurring mixtures thereof for controlling microbial growth.

[0002] In the production of cellulosic products such as cellulose, paper and board (hereinafter referred to as paper), cellulosic fibers are treated in aqueous suspensions under varying conditions. The amount of water in the production line is huge and the water is continuously recirculated in smaller or larger loops. The conditions in the production line are often susceptible to microbial growth. This is especially so during storage of the aqueous material.

[0003] For growth all microbes require that the system should include sufficient nutrients and that the pH, temperature, moisture, oxygen level etc. is adequate.

[0004] For breeding the microbes need time to grow and propagate in the system. The retention time must be long enough; otherwise, the cells will be washed out of the system. In a papermaking system the microbes can easily find locations with a sufficient retention time, e.g. water storage tanks, stock chests, treatment of broke and long pipelines.

[0005] Normal papermaking conditions are suitable for the growth of many kinds of microbes. The recirculating white water in the short and long circulation contains enough carbohydrates and other essential ingredients such as inorganics and trace elements. Also chemicals used in the papermaking themselves represent an ideal nutrient source, e.g. starch, or contain as impurities quite a lot of nutrient material, e.g. kaolin. The incoming raw water can also contain considerable amounts of nutrients.

[0006] The microbes found in a papermaking system can be divided into three main groups, bacteria, fungi and algae. The bacteria are either spore forming (anaerobic) or non spore forming (aerobic); the fungi comprise moulds and yeasts; and the prevailing algae are blue-green or green algae.

[0007] Many different kinds of problems can be caused by microbial growth, such as slime problems, runnability problems, corrosion problems, additive problems, and product problems.

[0008] Unless microbial control agents are added to the papermaking system, the general growth requirements for microbes are usually well satisfied in papermaking systems. Modern microbial control agents can roughly be divided into groups, which act in the following ways: oxidative decomposition of microbes (O_2 , ClO_2 , O_3 , peroxides); biocides, which inhibit or kill the microbes (organic, synthetic chemicals); and enzymes.

[0009] Oxygen is primarily used to prevent anaerobic conditions, while chlorine dioxide, ozone and peroxide work as biocides and disinfectants. They are quite effective but considered to be costly. Conventional biocides can be used alone or in combination with oxidative biocides. They are effective, but toxic. They can also be environmentally dangerous and hazardous for the working environment. A relatively new method of slime control is the use of enzyme. Enzymes are active at pH 3.5-10, which is an advantage compared with conventional biocides. However, enzymes have limited effect on bacteria.

[0010] Oxygen and oxygen-rich gases have primarily been used for preventing the formation of hydrogen sulfide and other volatile gases by anaerobic bacteria in waste waters. Robichaud, W. T., Tappi Journal, Feb. 1991, pp 149-153, has reported on the use of aeration for controlling anaerobic bacteria to improve product quality and mill safety in papermaking systems.

[0011] Use of carbon dioxide in paper making has been suggested in the prior art for various reasons mainly connected with specific needs for adjusting the pH or influencing the carbonate or bicarbonate chemistry. Examples of patents related to the use of carbon dioxide in papermaking systems are US 5,378,322 (Canadian Liquid Air); US 5,262,006 (Mo och Domsjö Ab); EP 0 296 198 (AGA Aktiebolag); EP 0 281 273 (The BOC Group); GB 2 008 562 (J. M. Voith GmbH); WO 99/24661 (AGA Aktiebolag); and WO 99/35333 (AGA Aktiebolag). None of these publications relate to the problem of microbial growth.

[0012] Mixtures of gases have been used in the packaging of foodstuffs in order to retain the foodstuff's original taste, texture and appearance. The gas mixtures usually consist of carbon dioxide, nitrogen and oxygen, but also other gases such as nitrous oxide, argon and hydrogen have been used. Carbon dioxide inhibits microbial activity on the foodstuff by reducing the pH and by penetrating biological membranes, causing changes in permeability and function. Nitrogen is primarily used to replace oxygen in packaging. Oxygen helps to preserve the oxygenated form of myoglobin, which gives meat its red colour. Oxygen is also required for fruit and vegetable respiration.

[0013] Amanatidou, A.; Smid, E. J.; Gorris, L. G.; J Appl Microbiol, March 1999, pp 429-38, have reported on the effect of elevated oxygen and carbon dioxide on the surface growth of vegetable-associated micro-organisms. Consistently strong inhibition was observed only when the two gases were used in combination.

[0014] As mentioned above, problems with microbial growth are conventional in papermaking systems, especially in storage tanks and in long pipe lines. Moreover, protection of the environment by the closing of the white water systems and increased recirculation of process waters as well as the increased use of waste paper has caused a marked increase in the microbial growth in the papermaking systems.

[0015] The papermaking industry consequently has an increasing need for means for reducing the microbial growth

in a technically feasible, inexpensive and environmentally friendly way.

[0016] It has now been found that a gaseous inhibitor in the form of carbon dioxide, nitrogen, a noble gas or a gas mixture containing one or more of said gases may be used for controlling microbial growth in a papermaking system.

[0017] Consequently, the present invention provides a process for controlling microbial growth in a production line for cellulosic products, which comprises providing an aqueous material containing water as well as suspended pulp fibers and/or additives therefor, maintaining said aqueous material under conditions susceptible to microbial growth, providing a gaseous inhibitor comprising a gas selected from carbon dioxide, nitrogen, noble gases and non-natural gas mixtures containing the same, and introducing said gaseous inhibitor to said aqueous material in an amount sufficient to significantly retard or inhibit the growth of microorganisms therein.

[0018] The gaseous inhibitor is preferably added immediately prior to and/or during storage of said aqueous material, since storage provides a sufficient time for the microbes to propagate.

[0019] The gaseous inhibitor should preferably consist of or contain a significant amount of carbon dioxide, nitrogen and/or a noble gas such as helium, neon, argon, krypton, xenon and/or radon. Carbon dioxide is the preferred gas according to the present invention. Among the noble gases argon is preferred.

[0020] If a gas mixture is used, said mixture should be a non-natural one. The gases of the mixture may be mixed before introduction into the aqueous material, or they may be added separately, simultaneously or sequentially. Although the gas mixture may contain oxygen, it should not have the composition of air, since air is known to inhibit only the growth of anaerobic microbes.

[0021] The gaseous inhibitor of the present invention may be used in combination with oxygen either by combining oxygen in the mixture of carbon dioxide, nitrogen and/or argon, or by adding oxygen separately from the other gases. When oxygen is added to the gas mixture, the amount of oxygen may vary from 10 to 90% of the total gas volume. However, according to another embodiment of the invention, an oxygen-rich gas is introduced into the aqueous material separately from the gaseous inhibitor of the present invention. Such an oxygen-rich gas may be added either before or after the addition of the inhibiting gas mixture.

[0022] The present invention will now be described in greater detail with reference to papermaking systems. It is, however, clear that the gaseous inhibiting system of the present invention may be used also in the production of cellulose, board, etc. As used in the context of the invention, a production line for cellulosic products comprises a line for the production of pulp, paper, board or the like. The production line will typically include at least a portion of reprocessed recovered paper and/or broke and will include loops of recirculating waters. The production line has a more or less closed water system, it being understood by those skilled in the art that the problems with microbial growth are prone to escalate in closed systems with an increasing and accumulating mass of microbes circulating in the system.

[0023] The temperature in the papermaking systems usually varies from 30 to 60 °C. Because of recycling, the temperature in the white water often exceeds 50 °C. Fungi and yeasts generally do not tolerate temperatures above 40 °C. Contrary to this, many bacteria thrive well in the high temperature range. pH usually varies from 3 to 10. Acidic conditions, pH 3-6, are very convenient for fungi and yeasts. Bacteria dominate under neutral and alkaline conditions, viz. at pH levels from 7 to 10. Anaerobic conditions can be found in many places throughout the production line, such as storage after dithionite bleaching, pulp chests and white water tanks.

[0024] Slime helps microbes to adhere onto surfaces and provides a food reserve. Microbial growth causes operating problems by plugging filters and screens, by reducing wire and felt life and by causing a decline of productivity due to breaks, wash-ups, etc. Corrosion induced by microbes is a consequence of vigorous microbial activity on surfaces.

[0025] The most important microbial species in this area include sulphate-reducing bacteria, which are anaerobic in character. There are, however, also a number of aerobic species which are harmful. Additives, such as starch, can deteriorate due to microbial activity, not to mention that a contaminated starch slurry can constitute a heavy inoculation of the white water system. When masses of microbes get loose from the actual growth place, the result may be seen as spots, holes or dirt specks in the final paper product. Spore-forming bacteria tolerate much heat and usually survive the drying stage. Thus they remain alive in the product and can be harmful later on.

[0026] In the working of the present invention note should be taken of the special circumstances of a papermaking system with its huge volumes of fluids, all the time on the move and having no definite surface where the microbes are prone to exist. This is in sharp contrast to, for instance, the packaging of food in protective atmospheres. The food moves nowhere within the package, its surface is solid and the gaseous atmosphere surrounds the product. In a papermaking system the aqueous material has a surface which changes continuously and it cannot be surrounded by the gas.

[0027] The term microbe or microorganism as used in the context of the present invention is intended to mean bacteria, fungi and/or algae such as described above. It should be understood that all of the microorganisms present in a papermaking system will not be influenced by the gaseous inhibitor of the present invention and that the gaseous inhibitor of the invention may therefore be used in combination with other inhibitors, such as oxygen-rich gases and biocides of various forms, as long as these do not interfere with the working of the invention itself.

[0028] According to the present invention microbial growth in the papermaking system is reduced by a gaseous

inhibitor. The gaseous inhibitor of the present invention is a gas or a gas mixture capable of inhibiting, partly or totally, the growth of microorganisms present in the papermaking system. The preferred single gas is carbon dioxide. The gaseous inhibitor may also comprise nitrogen and/or argon. Said gases may also be used alone, but they are more preferably used in combination with carbon dioxide. A suitable gaseous inhibitor consists of a mixture of carbon dioxide, nitrogen and argon. The mixture preferably contains at least 10% carbon dioxide.

[0029] The gaseous inhibitor may additionally contain oxygen. The oxygen should not be used in a combination resembling air, since such a mixture is effective only against anaerobic microorganisms. In a mixture of carbon dioxide and oxygen, the proportion of carbon dioxide should be between 90% and 10% and the proportion of oxygen should be between 10 % and 90%. It should be noted that normal air contains about 21 % oxygen and about 0.03% carbon dioxide.

[0030] A preferred embodiment of the invention comprises the use of a gaseous inhibitor consisting essentially of carbon dioxide, nitrogen, argon or mixtures thereof, which is introduced into a liquid flow of the aqueous material entering a storage tank for said aqueous material or into said storage tank itself. An oxygen-free gaseous inhibitor is preferably added in an amount sufficient to purge said aqueous material of oxygen and thereby inhibiting the growth of aerobic bacteria contained therein. Most aerobic bacteria are sensitive to the lack of oxygen and will eventually be killed off by such a procedure while the carbon dioxide will adversely affect many of the anaerobic species.

[0031] In an improvement of this embodiment, oxygen is used separately from the gaseous inhibitor of the present invention. Thus, after a suitable retention time the introduction of the gaseous inhibitor is followed by and/or preceded by an introduction of an oxygen containing gas into said storage tank in an amount sufficient to kill anaerobic bacteria in the aqueous material.

[0032] The introduction of the gaseous inhibitor and the introduction of oxygen may be repeated in an alternating manner during storage of said aqueous material.

[0033] The inhibitor is preferably added in a position where the risk for microbial growth is largest, i.e. in storage tanks for aqueous pulp suspensions or aqueous additives susceptible to microbial attack. However, the gaseous inhibitor may also be added to liquid flows of the aqueous material, to recirculating waters and to fresh water prior to its entering the system. The main principle of the present invention is to reduce the microbial growth at any position where it would otherwise rise to harmful proportions. It is not necessary to kill all the microbes but it is essential to reduce the microbial growth to such proportions that the harmful accumulations are minimized in the production line and in the final product.

[0034] In a preferred embodiment of the present invention the gaseous inhibitor is introduced into a liquid flow of the aqueous material or into a liquid flow of a diluent or additive for said material just prior to storage thereof. The gaseous inhibitor may also be introduced into any storage tank containing said aqueous material by bubbling the gas into the aqueous material and/or by filling the void space above the fluid.

[0035] In a typical process according to the present invention the aqueous material comprises a pulp suspension in a papermaking system and the suspension is treated with the gaseous inhibitor before it enters and/or as it is retained in a pulp storage tower, a stock chest, a broke tower or the like storage tank. The pulp suspension may also be stock in the stock preparation of a paper making system.

[0036] In a preferred embodiment the aqueous material to be treated comprises white water in a papermachine, preferably white water stored in the long circulation.

[0037] In another embodiment the aqueous material comprises a slurry of an additive chemical such as starch, coating, pigment, filler, or the like. Such additives are usually retained in aqueous suspension in readiness for use in the papermaking process and many of the additives contain nutrients making them susceptible to microbial attack. Typically this is true of starch, which in itself is a nutrient for many microorganisms. Many other additives, although inert in themselves, contain sufficient amounts of impurities to make them, with time, susceptible to microbial attacks. Treating such additive tanks with intermittent introductions of the gaseous inhibitor will effectively reduce the amount of microbes entering the system that way.

[0038] In a preferred embodiment of the present invention, the gaseous inhibitor is added at a late point, preferably just prior to the point where microbial attack is expected to be most severe, such as in a storage tower. Additional gaseous inhibitor (carbon dioxide/nitrogen/argon) should, if necessary, be added to the head space of the tower.

[0039] If oxygen is used in combination with the gaseous inhibitor, the oxygen should preferably be added directly after a pump feeding the aqueous material to the storage tower to make use of any turbulence to achieve a high rate of mixing. Additional oxygen should, if necessary be added to any of the tower's recirculation pipes to avoid creating any anaerobic areas in the storage tower.

[0040] It should be noted that although gases such as carbon dioxide and oxygen have previously been used in papermaking, the gaseous inhibitor of the present invention, comprising carbon dioxide, nitrogen or argon alone or in a non-natural gas mixture has not previously been used in papermaking systems for controlling microbial growth.

[0041] The aqueous materials of the cellulosic production line are processed to cellulosic products such as paper, board, dried pulp or the like material in a manner which is conventional in all other ways except for the biocidal treatment

of the present invention.

[0042] The present invention will now be illustrated with the following examples.

Example 1

[0043] A set of representative bacterial strains was isolated from a white water sample from a Swedish recycled pulp mill. The sample originally yielded 70 bacterial strains which each represented a group of different bacteria, which grew under similar conditions. The strains were tested on different media and their oxygen demands were studied. The results gave an indication of eight main groups. Further investigations showed that three out of the eight main groups seemed to be almost similar. Those three groups were put together to one large group.

[0044] From this dominating group, one strain was chosen for the carbon dioxide experiments. Two other strains from another recycled pulp mill were included in the experiments.

[0045] Glass bottles were tempered to 45 °C and CO₂ was added to create a carbon dioxide atmosphere. Bacteria from the three isolated strains were added to separate bottles together with a nutrient broth. A first sample representing "0 min" was taken out immediately from each bottle and then at a number of occasions during the next three hours. Each sample was diluted in dilution series with dilution 1/10 in six steps (10¹-10⁶) and grown on agar plates. The plates were incubated at 45 °C for two days, after which the number of colonies were counted and related to the dilution.

[0046] The percentage of surviving cells as a function of growth time under the influence of a CO₂ atmosphere is indicated in Table 1.

Table 1

Time (min)	Strain 1 (%)	Strain 2 (%)	Strain 3 (%)	Average (%)
0	100	100	100	100
10	75	141	92	103
20	34	76	77	62
30	73	137	58	89
40	50	48	47	48
50	61	94	36	64
60	59	46	67	57
80	39	88	44	57
100	16	55	42	38
120	18	43	16	47
140	5	35	7	16
160	4	41	4	16
180	9	24	2	12

[0047] The results clearly showed that after approximately 1 hour about 60% of the bacteria had survived, after 2 hours about 50% and after 3 hours slightly more than 10% (the values > 100% result from small mistakes in sampling or dilution).

Example 2

[0048] A mill produced wood-free paper from chemical pulp. At the time of the trials, the mill stored pulp for long periods of time in storage towers prior to the stock preparation. The storage caused problems with bad smell and black spots in the pulp, which was believed to be caused by high microbial activity during storage.

[0049] A full-scale trial of treatment with a gaseous inhibitor was performed. Thus, carbon dioxide gas was introduced in an amount of 1-2 kg CO₂/ton pulp just prior to the storage tower.

[0050] Both the pulp's bad smell and the number of black spots in the pulp were reduced. No negative effects of the CO₂ introduction were observed.

Example 3

[0051] A pulp storage tower having problems with an excess of microbial growth during a production stop is fed by a pump until the storage tower has been filled to about 80% of its capacity.

[0052] A gaseous inhibitor comprising a mixture of carbon dioxide/nitrogen/argon in the ratio 70/25/5 is added to the

feed line just before the pulp enters the tower. The gas is fed into the feed line at a rate of 1.5 kg gas per ton of pulp. The gas feeding is continued about 5 min after the feeding pump has stopped feeding pulp to the tower, in order to fill the head space of the tower with gaseous inhibitor.

[0053] 2 hours after the feeding of the gaseous inhibitor mixture, an oxygen-rich gas (air) is added into the pulp in the tower through a gas distribution tube. Addition of oxygen-rich gas is continued until a significant amount of oxygen is found to be present in the vent from the tower.

[0054] The following day the treatment is repeated by first feeding gaseous inhibitor of the present invention into the gas distribution tube, and after a residence time of about 2 hours, oxygen-rich gas is fed through the tube. The micro-biocidal treatment is repeated every day of the production stop.

[0055] The microbial growth in the storage tower is reduced to an acceptable level and at start-up no smell problems are encountered. A paper web is formed from the stored pulp in the normal way and only a minimal amount of dirt specks are seen in the paper.

Claims

1. A process for controlling microbial growth in a production line for cellulosic products comprising
 - providing an aqueous material containing water and suspended pulp fibers and/or additives therefor,
 - maintaining said aqueous material under conditions susceptible to microbial growth,
 - providing a gaseous microbe inhibitor selected from carbon dioxide, nitrogen, noble gases and non-natural mixtures containing the same,
 - introducing said gaseous microbe inhibitor to said aqueous material in an amount sufficient for said microbe inhibitor to significantly retard or inhibit the growth of micro-organisms therein.
2. A process according to claim 1, wherein said aqueous material is maintained in a storage vessel and said gaseous microbe inhibitor is introduced prior to and/or during said storage.
3. A process according to claim 1 or 2, wherein said gaseous microbe inhibitor consists essentially of carbon dioxide, nitrogen or one or more noble gases.
4. A process according to claim 1 or 2, wherein said noble gas is selected from helium, neon, argon, krypton, xenon and radon.
5. A process according to claim 1 or 2, wherein said gaseous microbe inhibitor is a mixture of carbon dioxide and nitrogen, preferably in a ratio of 10-90% carbon dioxide and 90-10% nitrogen.
6. A process according to claim 1 or 2, wherein said gaseous microbe inhibitor additionally contains oxygen.
7. A process according to claim 5, wherein said gaseous microbe inhibitor comprises a combination of carbon dioxide and oxygen, preferably in a ratio of 10-90% carbon dioxide and 90-10% oxygen.
8. A process according to claim 1, wherein said gaseous microbe inhibitor comprises a combination of carbon dioxide, nitrogen, noble gases and oxygen containing at least 10% carbon dioxide.
9. A process according to any one of claims 1 to 8, wherein said gaseous microbe inhibitor is introduced into a liquid flow of said material prior to said storage.
10. A process according to any one of claims 1 to 8, wherein said gaseous microbe inhibitor is introduced into a storage tank containing said aqueous material.
11. A process according to any one of claims 1 to 8, wherein said gaseous microbe inhibitor is introduced into a diluent for said material.
12. A process according to any one of claims 1 to 11, wherein said microorganisms comprise bacteria, fungi and/or

algae.

13. A process according to claim 1, wherein said gaseous microbe inhibitor, consisting essentially of carbon dioxide, nitrogen, argon gases or mixtures thereof, is introduced into a liquid flow entering a storage tank for said aqueous material or into said storage tank itself.

14. A process according to claim 13, wherein said introduction of gaseous microbe inhibitor is followed by and/or preceded by an introduction of an oxygen containing gas into said storage tank or into a liquid flow entering said storage tank.

15. A process according to claim 14, wherein said introduction of gaseous microbe inhibitor and said introduction of an oxygen containing gas are repeated in an alternating manner during storage of said aqueous material.

16. A process according to any one of claims 1 to 15, wherein said aqueous material is a pulp suspension located in a pulp storage tower, a stock chest, a broke tower or the like storage tank.

17. A process according to any one of claims 1 to 15, wherein said aqueous material is stock in the stock preparation of a paper making system.

18. A process according to any one of claims 1 to 15, wherein said aqueous material is white water in a papermachine, preferably white water in a white water tank of the long circulation.

19. A process according to any one of claims 1 to 15, wherein said aqueous material is a slurry of an additive chemical such as starch, coating, pigment, filler, or the like.

20. A process according to any one of claims 1 to 19, wherein said production line for cellulosic products comprises a line for the production of pulp, paper, board or the like.

21. A process according to claim 20, wherein said production line comprises the reprocessing of recovered paper and/or broke.

22. A process according to claim 20 or 21, wherein said production line has a substantially closed water system.

23. A process according to any one of claims 1 to 22, wherein said aqueous material treated with said gaseous microbe inhibitor is processed into a cellulosic product such as pulp, paper, board or the like.

24. Use of carbon dioxide, nitrogen or a noble gas alone or in a non-natural gas mixture as a gaseous microbe inhibitor for controlling microbial growth in an aqueous material containing water as well as suspended pulp fibers and/or additives therefor, which material is processed and/or stored in a production line for cellulosic products.

25. The use according to claim 24, wherein said gaseous microbe inhibitor consists of essentially carbon dioxide.

26. The use according to claim 25, wherein carbon dioxide and oxygen are used in combination or as alternating feeds to said material.

27. The use according to any one of claims 24 to 26 in the production of paper in a production line having a substantially closed water system and comprising the reprocessing of recovered paper.

Patentansprüche

1. Verfahren zur Kontrolle von mikrobiellem Wachstum in einer Herstellungsanlage für Zelluloseprodukte, umfassend

- Bereitstellen eines wässrigen Materials, enthaltend Wasser und suspendierte Zellstofffasern und/oder Zusätze dafür,
- Halten des wässrigen Materials unter für mikrobielles Wachstum anfälligen Bedingungen,
- Bereitstellen eines gasförmigen Mikrobeninhibitors, ausgewählt aus Kohlendioxid, Stickstoff, Edelgasen und nicht natürlichen Mischungen, die dieselben enthalten,

- Einführen des gasförmigen Mikrobeninhibitors in das wässrige Material in einer Menge, die ausreichend ist, damit der Mikrobeninhibitor das Wachstum von Mikroorganismen darin wesentlich verzögern oder hemmen kann.

- 5 2. Verfahren nach Anspruch 1, wobei das wässrige Material in einem Lagerungsbehälter gehalten wird und der gasförmige Mikrobeninhibitor vor und/oder während der Lagerung eingeführt wird.
3. Verfahren nach Anspruch 1 oder 2, wobei der gasförmige Mikrobeninhibitor im Wesentlichen aus Kohlendioxid, Stickstoff oder einem Edelgas bzw. mehreren Edelgasen besteht.
- 10 4. Verfahren nach Anspruch 1 oder 2, wobei das Edelgas aus Helium, Neon, Argon, Krypton, Xenon und Radon ausgewählt ist.
- 15 5. Verfahren nach Anspruch 1 oder 2, wobei der gasförmige Mikrobeninhibitor eine Mischung aus Kohlendioxid und Stickstoff vorzugsweise in einem Verhältnis von 10-90 % Kohlendioxid und 90-10 % Stickstoff ist.
6. Verfahren nach Anspruch 1 oder 2, wobei der gasförmige Mikrobeninhibitor zusätzlich Sauerstoff enthält.
- 20 7. Verfahren nach Anspruch 5, wobei der gasförmige Mikrobeninhibitor eine Kombination von Kohlendioxid und Sauerstoff vorzugsweise in einem Verhältnis von 10-90 % Kohlendioxid und 90-10 % Sauerstoff umfasst.
8. Verfahren nach Anspruch 1, wobei der gasförmige Mikrobeninhibitor eine Kombination von Kohlendioxid, Stickstoff, Edelgasen und Sauerstoff mit wenigstens 10 % Kohlendioxid umfasst.
- 25 9. Verfahren nach einem der Ansprüche 1 bis 8, wobei der gasförmige Mikrobeninhibitor in einen flüssigen Strom des Materials vor der Lagerung eingeführt wird.
10. Verfahren nach einem der Ansprüche 1 bis 8, wobei der gasförmige Mikrobeninhibitor in einen das wässrige Material enthaltenden Lagertank eingeführt wird.
- 30 11. Verfahren nach einem der Ansprüche 1 bis 8, wobei der gasförmige Mikrobeninhibitor in ein Verdünnungsmittel für das Material eingeführt wird.
- 35 12. Verfahren nach einem der Ansprüche 1 bis 11, wobei die Mikroorganismen Bakterien, Pilze und/oder Algen umfassen.
- 40 13. Verfahren nach Anspruch 1, wobei der gasförmige Mikrobeninhibitor, bestehend im Wesentlichen aus Kohlendioxid-, Stickstoff-, Argongasen oder Mischungen davon, in einen flüssigen Strom, der in einen Lagertank für das wässrige Material hinein läuft, oder in den Lagertank selber eingeführt wird.
- 45 14. Verfahren nach Anspruch 13, wobei der Einführung des gasförmigen Mikrobeninhibitors eine Einführung eines sauerstoffhaltigen Gases in den Lagertank oder in einen flüssigen Strom, der in einen Lagertank für das wässrige Material hinein läuft, folgt und/oder vorausgeht.
- 50 15. Verfahren nach Anspruch 14, wobei die Einführung des gasförmigen Mikrobeninhibitors und die Einführung eines sauerstoffhaltigen Gases während der Lagerung des wässrigen Materials abwechselnd wiederholt werden.
16. Verfahren nach einem der Ansprüche 1 bis 15, wobei das wässrige Material eine Zellstoffsuspension ist, die sich in einem Zellstofflagerturm, einer Stoffbütte, einem Ausschuss-Turm oder einem ähnlichen Lagertank befindet.
- 55 17. Verfahren nach einem der Ansprüche 1 bis 15, wobei das wässrige Material ein Stoff bei der Stoffaufbereitung eines Papierherstellungssystems ist.
18. Verfahren nach einem der Ansprüche 1 bis 15, wobei das wässrige Material Rückwasser in einer Papiermaschine ist, vorzugsweise Rückwasser in einem Rückwassertank des langen Kreislaufs.
19. Verfahren nach einem der Ansprüche 1 bis 15, wobei das wässrige Material eine Schlämme einer Zusatzmittelchemikalie wie beispielsweise Stärke, Beschichtung, Pigment, Füllmittel oder dergleichen ist.

20. Verfahren nach einem der Ansprüche 1 bis 19, wobei die Herstellungsanlage für Zelluloseprodukte eine Anlage für die Herstellung von Zellstoff, Papier, Pappe oder dergleichen umfasst.

21. Verfahren nach Anspruch 20, wobei die Herstellungsanlage die Wiederverarbeitung von wiedergewonnenem Papier und/oder Ausschuss umfasst.

22. Verfahren nach Anspruch 20 oder 21, wobei die Herstellungsanlage ein im Wesentlichen geschlossenes Wassersystem hat.

23. Verfahren nach einem der Ansprüche 1 bis 22, wobei das mit dem gasförmigen Mikrobeninhibitor behandelte, wässrige Material zu einem Zelluloseprodukt wie beispielsweise Zellstoff, Papier, Pappe oder dergleichen verarbeitet wird.

24. Verwendung von Kohlendioxid, Stickstoff oder einem Edelgas alleine oder in einer nicht natürlichen Gasmischung als ein gasförmiger Mikrobeninhibitor zur Kontrolle von mikrobiellem Wachstum in einem Wasser sowie suspendierte Zellstofffasern und/oder Zusatzmittel dafür enthaltenden, wässrigen Material, wobei das Material in einer Herstellungsanlage für Zelluloseprodukte verarbeitet und/oder gelagert wird.

25. Verwendung nach Anspruch 24, wobei der gasförmige Mikrobeninhibitor im Wesentlichen aus Kohlendioxid besteht.

26. Verwendung nach Anspruch 25, wobei Kohlendioxid und Sauerstoff in Kombination verwendet oder dem Material abwechselnd zugeführt werden.

27. Verwendung nach einem der Ansprüche 24 bis 26 in der Papierherstellung in einer Herstellungsanlage mit einem im Wesentlichen geschlossenen Wassersystem und umfassend die Wiederverarbeitung von wiedergewonnenem Papier.

Revendications

1. Procédé de contrôle de la croissance microbienne dans une chaîne de production de produits cellulosiques, comprenant :

- la fourniture d'un matériau aqueux contenant de l'eau et des fibres de pâte en suspension et/ou des additifs pour celles-ci,
- le maintien dudit matériau aqueux dans des conditions susceptibles d'une croissance microbienne,
- la fourniture d'un inhibiteur de microbes gazeux choisi parmi le dioxyde de carbone, l'azote, les gaz nobles et des mélanges non naturels contenant ceux-ci,
- l'introduction dudit inhibiteur de microbes gazeux dans ledit matériau aqueux en une quantité suffisante pour que ledit inhibiteur de microbes retarde ou inhibe de façon significative la croissance de micro-organismes dans celui-ci.

2. Procédé selon la revendication 1, dans lequel ledit matériau aqueux est maintenu dans un récipient de stockage et ledit inhibiteur de microbes gazeux est introduit préalablement audit et/ou pendant ledit stockage.

3. Procédé selon la revendication 1 ou 2, dans lequel ledit inhibiteur de microbes gazeux est constitué essentiellement de dioxyde de carbone, d'azote ou d'un ou plusieurs gaz nobles.

4. Procédé selon la revendication 1 ou 2, dans lequel ledit gaz noble est choisi parmi l'hélium, le néon, l'argon, le krypton, le xénon et le radon.

5. Procédé selon la revendication 1 ou 2, dans lequel ledit inhibiteur de microbes gazeux est un mélange de dioxyde de carbone et d'azote, préférablement en un rapport de 10-90% de dioxyde de carbone et de 90-10% d'azote.

6. Procédé selon la revendication 1 ou 2, dans lequel ledit inhibiteur de microbes gazeux contient en outre de l'oxygène.

EP 1 287 201 B1

7. Procédé selon la revendication 5, dans lequel ledit inhibiteur de microbes gazeux comprend une association de dioxyde de carbone et d'oxygène, préférablement en un rapport de 10-90% de dioxyde de carbone et de 90-10% d'oxygène.
- 5 8. Procédé selon la revendication 1, dans lequel ledit inhibiteur de microbes gazeux comprend une association de dioxyde de carbone, d'azote, de gaz nobles et d'oxygène, contenant au moins 10% de dioxyde de carbone.
9. Procédé selon l'une quelconque des revendications 1 à 8, dans lequel ledit inhibiteur de microbes gazeux est introduit dans un courant liquide dudit matériau préalablement audit stockage.
- 10 10. Procédé selon l'une quelconque des revendications 1 à 8, dans lequel ledit inhibiteur de microbes gazeux est introduit dans un bassin de stockage contenant ledit matériau aqueux.
11. Procédé selon l'une quelconque des revendications 1 à 8, dans lequel ledit inhibiteur de microbes gazeux est introduit dans un diluant pour ledit matériau.
- 15 12. Procédé selon l'une quelconque des revendications 1 à 11, dans lequel lesdits micro-organismes comprennent les bactéries, les champignons et/ou les algues.
- 20 13. Procédé selon la revendication 1, dans lequel ledit inhibiteur de microbes gazeux, constitué essentiellement de dioxyde de carbone, d'azote, de gaz d'argon ou de mélanges de ceux-ci, est introduit dans un courant liquide pénétrant dans un bassin de stockage pour ledit matériau aqueux ou dans ledit bassin de stockage lui-même.
- 25 14. Procédé selon la revendication 13, dans lequel ladite introduction d'inhibiteur de microbes gazeux est suivie et/ou précédée d'une introduction d'un gaz contenant de l'oxygène dans ledit bassin de stockage ou dans un courant liquide pénétrant dans ledit bassin de stockage.
- 30 15. Procédé selon la revendication 14, dans lequel ladite introduction d'inhibiteur de microbes gazeux et ladite introduction de gaz contenant de l'oxygène sont répétées de manière alternative au cours du stockage dudit matériau aqueux.
- 35 16. Procédé selon l'une quelconque des revendications 1 à 15, dans lequel ledit matériau aqueux est une suspension de pâte située dans une tour de stockage de pâte, dans un cuvier de pâte, une tour de cassé de fabrication ou un bassin de stockage semblable.
- 40 17. Procédé selon l'une quelconque des revendications 1 à 15, dans lequel ledit matériau aqueux est de la pâte liquide dans le traitement de la pâte d'un système de fabrication de papier.
18. Procédé selon l'une quelconque des revendications 1 à 15, dans lequel ledit matériau aqueux est de l'eau collée dans une machine à papier, préférablement de l'eau collée dans un réservoir à eau collée de la circulation longue.
- 45 19. Procédé selon l'une quelconque des revendications 1 à 15, dans lequel ledit matériau aqueux est une bouillie d'un additif chimique, tel que l'amidon, un revêtement, un pigment, une charge, ou similaire.
20. Procédé selon l'une quelconque des revendications 1 à 19, dans lequel ladite chaîne de production pour les produits cellulosiques comprend une chaîne pour la production de pâte, de papier, de carton ou similaire.
21. Procédé selon la revendication 20, dans lequel ladite chaîne de production comprend le retraitement de papier et/ou de cassé de fabrication récupérés.
- 50 22. Procédé selon la revendication 20 ou 21, dans lequel ladite chaîne de production a un système d'eau sensiblement fermé.
23. Procédé selon l'une quelconque des revendications 1 à 22, dans lequel ledit matériau aqueux traité par ledit inhibiteur de microbes gazeux est traité en un produit cellulosique tel que la pâte, le papier, le carton ou similaire.
- 55 24. Utilisation de dioxyde de carbone, d'azote ou d'un gaz noble seul ou dans un mélange gazeux non naturel comme inhibiteur de microbes gazeux pour le contrôle de la croissance microbienne dans un matériau aqueux contenant

EP 1 287 201 B1

de l'eau ainsi que des fibres de pâte en suspension et/ou des additifs pour celles-ci, lequel matériau étant traité et/ou stocké dans une chaîne de production pour les produits cellulosiques.

5 **25.** Utilisation selon la revendication 24, dans laquelle ledit inhibiteur de microbes gazeux est constitué essentiellement de dioxyde de carbone.

26. Utilisation selon la revendication 25, dans laquelle du dioxyde de carbone et de l'oxygène sont utilisés en association ou comme alimentations alternées audit matériau.

10 **27.** Utilisation selon l'une quelconque des revendications 24 à 26 dans la production de papier dans une chaîne de production ayant un système d'eau sensiblement fermé, et comprenant le retraitement de papier récupéré.

15

20

25

30

35

40

45

50

55