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(54) Title: COMPOSITION FOR BLEACHING WOODPULP AND PROCESS FOR ITS PRODUCTION

(57) Abstract

A composition for bleaching woodpulp comprising one or more optical bleaching agents and another additive ingredient selected from the group consisting of caseine, caseine derivatives, soya proteins and their mixtures in order to obtain paper products with high amounts of woodpulp with respect to prior art products and having improved touch and visual features. Said composition can also be used for bleaching all substrates with a scarce white degree during the production and/or the treatment of the substrates; such as not bleached celluloses, semibleached celluloses or finished paper products. A process for producing the composition of the invention is also described.

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"COMPOSITION FOR BLEACHING WOODPULP AND PROCESS FOR ITS PRODUCTION"

The present invention relates to a composition for bleaching woodpulp in order to obtain paper products having improved visual and touch features.

It is known that white paper forms most of paper production and that white, monolayer or multilayer board forms almost the totality of board production.

In order to obtain a high white degree of paper and board, white celluloses and/or extra-white celluloses and pure white mineral fillers such as caolin, calcium carbonate and so forth are used by paper industry. The white degree so obtained by the use of these selected raw materials can be further increased by using said optical bleaching agents.

Optical bleaching agents are fluorescent substances able to absorb UV radiation and to emit light with lower energy and, thereafter, with higher wavelength in the visible range of the solar spectrum corresponding to the blue-violet wavelength. In this way, when the same amount of incident light is absorbed, the reflected visible light increases, thereby the sheet looks whiter to the naked eye than what it actually is. This is allowed by the compensation of the blue-violet emission of the optical bleaching agent towards the yellowish component of paper, thereby the whole effect is due to the mixing of substrate colour with the fluorescent light.

It is therefore obvious that the characteristic colour of the substrate strongly affects the final bleaching result. The optical bleaching agent efficiency is, as a consequence, always related to the material to be bleached.

The two basic components used in paper industry are several kinds of celluloses and woodpulps, the quantitative ratios of which determine the preparation of different kinds of paper products, which are available on the market.

Different aqueous compositions of optical bleaching agents, aimed to bleach celluloses, which initially already have a rather low yellowish component, are known.

The woodpulps on the market are identified by codes which state the production process of woodpulp itself and have their own basic peculiar characteristic brighteness as stated in the following:

WOODPULP	BRIGHTENESS (%) ISO
SGW (Stone Ground Wood)	63

RMP (Refiner Mechanical Pulp)	62
TMP (ThermoMechanical Pulp)	60
PGW (Pressure Ground Wood)	58
TGW (ThermoGrinding Wood)	56
CTMP (ChemoThermoMechanical Pulp)	60
CMP (ChemoMechanical Pulp)	52

During the paper production process, because of the strongly yellow peculiarity of the different woodpulp produced, either a reductive bleaching with sodium dithionite ($Na_2S_2O_4$), leading to an increase of white degree up to 12% ISO or an oxidative bleaching with sodium peroxide or hydrogen peroxide at pH 10-10.5, leading to an increase of white degree up to 14%, is usually carried out. A combination of the two bleaching processes in sequence peroxide/dithionite is usually used in order to achieve a brighteness increase of 16-20 points compared to the initial brightness stated above.

The woodpulps so bleached cannot be further bleached to a higher white degree, even by using a high concentration of optical bleaching agent and, besides, their yellowing tendency cannot be changed ("ULMANN'S ENCYCLOPEDIA OF INDUSTRIAL CHEMISTRY", 1991, vol. A18, pg. 601-602).

Moreover woodpulps have also scarce touch features, i.e. the surface of the sheets obtained with high amounts of woodpulp is rough, thereby, it is not very satisfactory to touch, contrary to sheets obtained with high amounts of cellulose, which have smooth surfaces.

Up to now, in spite of the efforts made, a method enabling the woodpulps to have visual features similar to the cellulose has not been found out as yet.

It is further known that woodpulp is a fibrous product which is combined with the cellulose in order to give a stiffness degree to the final paper product. Such feature is a cubic function of the material thickness, and as woodpulp density is lower than that of cellulose, being the weight equal, the woodpulp is thicker and stiffer than the cellulose. The woodpulp also has a higher opacity degree and remarkably lower costs up to 25-30% less than the costs of cellulose.

It is hence obvious that if more woodpulp could be used instead of cellulose for the production of the final paper, a lighter product having the same stiffness, opacity and

brightness features would be obtained at lower costs than prior art products. It must furthemore be taken into account that a decrease in cellulose use would reduce the environmental problem of tree felling considering that the yield of woodpulp is about 90 % and of cellulose is about 55%.

The advantage, which would be achieved by having a composition allowing the bleaching problem of woodpulps to be solved and the remarkable use in place of the cellulose, is therefore clear.

It is an object of the present invention to provide a composition for bleaching the woodpulp in order to obtain light, stiff and mat paper products having a high white degree and improved touch features.

It is further an object of the present invention to provide a composition for bleaching the woodpulp in order to reduce the use of the cellulose in paper industry.

It is still another object of the present invention to provide compositions that allow the production of paper products at lower costs.

Aforesaid objects and advantages are achieved by using a composition as stated in claim 1.

The additive ingredient which is added to the optical bleaching agents in order to obtain the bleaching composition according to the present invention can be caseine, caseine derivatives, soya proteins or their mixtures.

The composition according to the invention will be better understood from the following detailed description followed by two illustrative and not limitative preferred embodiments of the invention. Wherever the term "parts" is used, it means "parts by weight", unless otherwise specified.

The woodpulp can be any woodpulp however obtained, especially selected from the group consisting of SGW (Stone GroundWood), RMP (Refined Mechanical Pulp), TMP (ThermoMechanical Pulp), PGW (Pressure Ground Wood), TGW (ThermoGrinding Wood), CTMP (ChemoThermoMechanical Pulp), CMP (ChemoMechanical Pulp) and preferably from poplar and fir-tree.

The woodpulp to be treated with the composition of the invention is preferably either CTMP, which is obtained by a mechanical process along with chemothermal treatment, or CMP, which is obtained by mechanical process together with treatments with alkali compounds or acids.

The composition according to the invention can be directly applied on the obtained woodpulp or preferably on the woodpulp pretreated with bleaching cycles, which are of generally known industrial kind and preferably cycles of peroxide/sodium dithionite.

The additive ingredient is dissolved in an alkaline solution at a concentration between 1% and 30% based on weight solution, and preferably 13% of caseine or derivative thereof or 16% of soya proteins. Said alkaline solution of the additive ingredient is added to the optical bleaching agent solution at ratios of additive ingredient solution to optical bleaching agent solution between 0.1 and 10, preferably 0.5-5 and more preferably equal to 1. The optical bleaching agent can be added as a powder or more preferably it can be dissolved in water.

Said optical bleaching agent is a derivative of compounds selected from the group consisting of carbocycles, distyrilbenzenes, distyrilbiphenyls, divinylstilbenes, bistriazinylaminostilbenes, stilbenyl-2H-triazoles, stilbenyl-2H-naphtho[1,2-d]triazoles, bis(1,2,3-triazol-3-yl)stilbenes, benzoxazoles, stilbenylbenzoxazoles, bis(benzoxazoles), furans, benzo[b]furans, benzimidazoles, bis(benzo[b]furan-2-yl)biphenyls,cationic benzimidazoles, 1,3-diphenyl-2-pyrazolines, coumarins, naphthalimides, 1,3,5-triazin-2-yl derivatives and mixtures thereof. Said optical bleaching agent is preferably a compound derived from the family of stilbenes and more preferably is a derivative of bistriazinylaminostilbene. Therefore the optical bleaching agent can be directly added to the caseine or a derivative thereof solution without any pre-treatment.

The caseine derivatives are preferably caseinates of different alkaline or earth alkaline metals, such as e.g. sodium caseinate, and these are dissolved before adding the optical bleaching agent. According to the invention it is provided that a mixture of caseine and a derivative thereof is used under the same conditions stated for the single components.

The solution of additive ingrendient and optical bleaching agent is mixed at a temperature between 15 °C and 80 °C, preferably 30-50 °C and more preferably 40 °C for about 10-30 minutes. To said composition optional excipients such as preservatives, pH stabilizers are added in order to have a formulation to be placed on the market as a ready for use product. On the other hand the composition can be prepared when being used.

The composition according to the invention is metered on the base of the amount of dry fiber to be bleached and on the base of the desired white degree. It is preferably added in amounts between 0.1 and 10% based on the fiber dry weight to be bleached.

The maximum white degree achievable by applying the composition of the invention is obtained by treating the material to be bleached at pH ≤ 7 after it has already been treated with the composition of the invention, preferably at pH 5.5, with a compound selected from the group consisting of aluminum salts, mineral acids, organic acids, preferably aluminum sulfate.

The composition according to the invention can, of course, be used on any material having a scarce "basic white", therefore on non bleached, semibleached celluloses, semicelluloses or can be also used in any bleaching treatment on paper substrates, producing the same effects of the application on woodpulp.

Example 1

100 parts of caseine are dispersed in 400 parts of water by stirring for ten minutes. Thereafter 8 parts of sodium tetraborate and 100 parts of water are added and the solution is heated at a temperature of 40 °C for 10 minutes. 7 parts of an alkali, preferably ammonia, are then added, heating at 60 °C and stirring for further 20 minutes. Finally, 160 parts of water are added and the solution is stirred for 20 minutes at 60 °C.

50 parts of the solution so obtained are then mixed with 50 parts of a liquid optical bleaching agent or a powder optical bleaching agent dissolved in water so as to have a solution with a maximal possible concentration. The product so obtained can be used directly or be added with optional additives for the preparation of a ready-to-sell product.

Example 2

130 parts of soya proteins are dispersed in 500 parts of water by stirring for ten minutes. Thereafter 10 parts of sodium tetraborate and 100 parts of water are added and the solution is heated at a temperature of 40 °C for 10 minutes. 10 parts of an alkali, preferably ammonia, are then added, heating at 60 °C and stirring for further 20 minutes. Finally, 200 parts of water are added and the solution is stirred for 20 minutes at 60 °C.

50 parts of the solution so obtained are then mixed with 50 parts of a liquid optical bleaching agent or a powder optical bleaching agent dissolved in water so as to have a solution with a maximal possible concentration. The product so obtained can be used directly or be added with optional additives for the preparation of a ready-to-sell product.

The surprising advantages of the composition of the invention are now illustrated with reference to the results of tests carried out in laboratory, at the Experimental Centre for Cellulose, Paper and textile, vegetable and artificial Fibers in Milan.

Reflection Test 1

4 samples were examined as stated in the following:

Sample 1: woodpulp sheet, pretreated with bleaching cycles, manufactured with a static sheetmaker (FS) with grammage of about 120 g/m^2

Sample 2: woodpulp sheet as sample 1 treated with 2% of optical bleaching agent and caseine solution at 1:1 ratio based on woodpulp dry weight.

Sample 3: sheet of bleached cellulose having short fibers.

Sample 4: woodpulp sheet, pretreated with bleaching cycles, manufactured by dynamic sheetmaker (Fd) of TechPap with a grammage of 120g/m^2 treated with 2% of optical bleaching agent and caseine solution at 1:1 ratio based on woodpulp dry weight.

These four samples were tested for the white degree through reflection according to the UNI 7623-86 method. Data which is set forth in Table 1 were obtained.

Table 1

Sample	1	2	3	4
Reflection (%) ISO	77.0	84.4	83.4	84.9

Sample 2 treated with the composition according to the invention shows a higher reflection percentage than sample 3, made of bleached cellulose having short fibers. Sample 4 shows results almost equal to sample 2, underlining that the surprising white effect is feasible on industrial scale, being sample 4 obtained through a dynamic system, which simulates the industrial conditions of making the sheet.

Reflection test 2

7 samples, four of which stated as A and being of CTMP woodpulp of Miller Western S.W., and the remaining three, stated as B and being of P85 CMP woodpulp of Sicem Saga, were examined. Both woodpulps had been pretreated with bleaching cycles of peroxide/sodium dithionite.

Sample 1A: woodpulp A

Sample 2A: woodpulp A treated with 1% of a optical bleaching agent available on the market

Sample 3A: woodpulp A treated with 2% of the solution of the invention at optical bleaching agent/caseine ratio equal to 1.

Sample 4A: woodpulp A treated with 4% of the solution of the invention at optical bleaching agent/caseine ratio equal to 1.

Sample 1B: woodpulp B

Sample 2B: woodpulp B treated with 1% of a optical bleaching agent available on the market.

Sample 3B: woodpulp B treated with 2% of the solution of the invention at optical bleaching agent/caseine ratio equal to 1.

The reflection was tested in order to establish the white degree according to the UNI 7623-86 method, and the data thereof are set forth in Table 2.

Table 2

Sample	1A	2A	3A	4A	1B	2B	3B
Reflection (%) ISO	72.7	73.4	77.9	79.1	78.6	81.9	86.6

As shown from data in Table 2 the reflection remarkably increases for sample 3A and still more by doubling the amount of the solution (4A) according to the invention in comparison with sample (1A) which is not treated or sample (2A) treated with a optical bleaching agent. Sample 1B of woodpulp with a reflection higher than sample A, when treated with the composition (3B) of the invention, shows the same increase of reflection like 3A, confirming the remarkable bleaching result of the composition according to the invention.

Reflection test 3

Reflection test 1 was repeated using the same woodpulp bleached under the same conditions and amounts, except for caseine which was replaced by soya proteins. Reflection data (%ISO) are set forth in the following Table 3.

Table 3

Sample	1	2	3	4
Reflection % ISO	77.0	84.0	83.4	84.2

As indicated in Table 3, samples 2 and 4, treated with the solution of the invention, show higher reflection than sample 3, being of bleached cellulose with short fibers, confirming the observations already made in the case of reflection test 1 which uses caseine.

It is obviuos from the previous detailed description and preferred embodiments that modifications and/or substitutions with functionally equivalent parts, such as replacement of the ingredient additive of the invention with functionally similar proteic substances, can be made without departing from the scope of the appended claims.

CLAIMS

1. Composition for bleaching woodpulp comprising one or a mixture of optical bleaching agents characterized by comprising a further additive ingredient selected from the group consisting of caseine, caseine derivatives, soya proteins and mixtures thereof.

2. Composition of claim 1 characterized in that the woodpulp is selected from the group consisting of SGW (Stone GroundWood), RMP (Refined Mechanical Pulp), TMP (ThermoMechanical Pulp), PGW (Pressure Ground Wood), TGW (ThermoGrinding Wood), CTMP (ChemoThermoMechanical Pulp), CMP (ChemoMechanical Pulp).

3. Composition of claim 2, characterized in that the woodpulp is CTMP (ChemoThermoMechanical Pulp) and CMP (ChemoMechanical Pulp).

4. Composition of any one of the preceding claims wherein the woodpulp has been pre-treated with bleaching cycles.

5. Composition according to claim 4 wherein the bleaching cycles of the pre-treatment of the woodpulp are cycles of peroxide/sodium dithionite.

6. Composition according to any one of the preceding claims wherein the additive ingredient is dissolved in a solution at a concentration between 1% and 30% based on weight solution.

7. Composition according to claim 6 wherein said additive ingredient is caseine and/or a derivative thereof dissolved in a solution at a concentration of 13% based on weight solution.

8. Composition according to claim 6 wherein said additive ingredient is soya proteins at a concentration of 16% based on weight solution.

9. Composition according to any one of the preceding claims characterized in that the ratio of additive ingredient solution to optical bleaching agent solution is from 0.1 to 10, preferably 0.5-5 and more preferably equal to 1.

10. Composition according to any one of the preceding claims wherein the optical bleaching agent is a derivative of compounds selected from the group consisting of carbocycles, distyrilbenzenes, distyrilbiphenyls, divinylstilbenes, bistriazinylaminostilbenes, stilbenyl-2H-triazoles, stilbenyl-2H-naphtho[1,2-d]triazoles, bis(1,2,3-triazol-3-yl)stilbenes, benzoxazoles, stilbenylbenzoxazoles, bis(benzoxazoles), furans, benzo[b]furans, benzimidazoles, bis(benzo[b]furan-2-yl)biphenyls,cationic benzimidazoles, 1,3-diphenyl-2-pyrazolines, coumarins, naphthalimides, 1,3,5-triazin-2-yl derivatives and mixtures thereof.

11. Composition of claim 10 wherein the optical bleaching agent is a derivative from the family of stilbenes, preferably a derivative of bistriazinylaminostilbene.

12. Composition of any one of the preceding claims wherein the derivative of caseine is caseinate of alkaline or earth alkaline metals.

13. Composition of claim 12 wherein the derivative of caseine is sodium caseinate.

14. Composition according to any one of the preceding claims characterized in that said composition is added with excipients for the preparation of a commercial ready-to-sell product.

15. Process for producing the composition according to claim 1, characterized by the following steps:

a) dissolving the additive ingredient selected from the group consisting of caseine, caseine derivatives, soya proteins and mixtures thereof;

b) heating the solution so obtained at a temperature between 15 °C and 80 °C and stirring for 10-30 minutes;

c) adding an alkali while heating at a temperature between 40 and 80 °C and stirring for 10-30 minutes;

d) adding water and stirring for 10-30 minutes by heating at a temperature between 40 and 80 °C; and

e) combining the solution of step d) with the optical bleaching agent solution.

16. Process according to claim 15 wherein the optical bleaching agent of step e) is either already ready-to-use or a water solution of optical bleaching agent powder prepared when it is used.

17. Process according to claims 15-16 comprising a further treatment step of the material, already bleached with the composition prepared according to the process, at pH below or equal to 7, preferably 5.5, with a compound selected from the group consisting of aluminum salts, mineral acids, organic acids, preferably aluminum sulfate.

18. Use of the composition according to any one of claims1-17 for bleaching woodpulp for obtaining paper products with high white degree.

19. Use of the composition according to any one of claims 1-17 for bleaching non bleached, semibleached celluloses or semicelluloses for obtaining paper products with high white degree.

20. Use of the composition according to any one of claims 1-17 for bleaching

treatment of finished paper products.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C08L89/00 D21C9/00 D21H21/30

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 D21C C08L D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 14 69 229 A (GENERAL ANILINE & FILM CORP.) 27 November 1969 see page 2, paragraph 2 see page 5, line 5 - page 8, line 9 ---	1-7, 9-11,14, 18-20
X	DE 37 37 553 A (GÖPPINGER KALIKO GMBH) 18 May 1989 see claim 9 ---	1-6,8,9, 14
X	GB 1 021 887 A (BADISCHE ANILIN & SODA-FABRIK AKTIENGESELLSHAFT) 9 March 1966 see page 1, line 20 - line 25 see page 3, line 14 - line 85 --- -/--	1-20

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate. of the relevant passages	Relevant to claim No.
A	DE 42 02 703 A (BAYER AG) 5 August 1993 see the whole document	1-7, 9-11,14, 18-20
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Information on patent family members

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