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(72) Inventeurs/Inventors:
RUF, HARTMUT, AT;
EICHINGER, DIETER, AT;
SCHILD, GABRIELE, AT;
FEILMAIR, WILHELM, AT

(73) Propriétaire/Owner:
LENZING AKTIENGESELLSCHAFT, AT

(74) Agent: SIM & MCBURNEY

(54) Titre : CORPS MOULES EN CELLULOSE ET PROCEDE DE PRODUCTION CORRESPONDANT
(54) Title: CELLULOSE MOULDED BODY AND PROCESS FOR ITS PRODUCTION

(57) Abrégé/Abstract:
The invention is concerned with a process for the production of cellulose moulded bodies exhibiting high brightness, which is characterized by a combination of the steps of - bleaching pulp using a bleaching agent, provided that the bleaching agent does not contain chlorine or any chlorine compound, - dissolving the pulp bleached using the bleaching agent in an aqueous tertiary amine-oxide, a mouldable cellulose solution being obtained, and - processing the mouldable cellulose solution into moulded bodies.

Abstract

The invention is concerned with a process for the production of cellulose moulded bodies exhibiting high brightness, which is characterized by a combination of the steps of

- bleaching pulp using a bleaching agent, provided that the bleaching agent does not contain chlorine or any chlorine compound,
- dissolving the pulp bleached using the bleaching agent in an aqueous tertiary amine-oxide, a mouldable cellulose solution being obtained, and
- processing the mouldable cellulose solution into moulded bodies.

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CELLULOSE MOULDED BODY AND PROCESS FOR ITS PRODUCTION

The invention is concerned with a cellulose moulded body, particularly a cellulose fibre, and a process for its production.

For some decades there has been searched for processes for the production of cellulose fibres able to substitute the viscose process, today widely employed. As an alternative which is interesting for its reduced environmental impact among other reasons, it has been found to dissolve cellulose without derivatisation in an organic solvent and extrude from this solution moulded bodies, e.g. fibres, films and other moulded bodies. Fibres thus extruded have received by BISFA (The International Bureau for the Standardization of man made fibers) the generic name Lyocell. By an organic solvent, BISFA understands a mixture of an organic chemical and water.

It has turned out that as an organic solvent, a mixture of a tertiary amine-oxide and water is particularly appropriate for the production of cellulose moulded bodies. As the amine-oxide, primarily N-methylmorpholine-N-oxide (NMMO) is used. Other amine-oxides are described e.g. in EP-A - 0 553 070. A process for the production of mouldable cellulose solutions is known e.g. from EP-A - 0 356 419. The production of cellulose moulded bodies using tertiary amine-oxides generally is referred to as amine-oxide process.

In EP-A - 0 356 419, a process for the production of spinnable cellulose solutions is described, wherein as a starting material, among other substances, a suspension of cellulose in liquid, aqueous N-methylmorpholine-N-oxide (NMMO) is used. This process consists in transforming the suspension in a thin-film treatment apparatus in one single step and continuously into a mouldable solution. Finally, the mouldable solution is spun into filaments by means of a forming tool such as a spinneret, which filaments are conducted across an air gap, wherein they are stretched, into

a precipitation bath, wherein the cellulose is precipitated. Afterwards, the cellulose fibres obtained are washed.

To avoid an undesired discolouration of the fibres, primarily due to lignin and coloured organic compounds, it is known to bleach the pulp before processing it. For this purpose, raw pulp is reacted with a number of bleaching agents such as elemental chlorine, chlorine compounds such as hypochlorite and chlorine dioxide, oxygen, peroxide and ozone, under exactly defined conditions, said reaction being carried out using a combination of these chemicals in a certain order, which is referred to as bleaching sequence.

Classical bleaching processes start using elemental chlorine, which chlorinates or oxidizes to different extents depending on the pH value. Hypochlorite and chlorine dioxide oxidize the lignin and coloured compounds. Between each of the reaction steps of these bleaching agents, usually an alkaline extraction is provided to remove the lignin brought into solution and the other compounds from the reaction medium. For an overview of the technique of pulp bleaching R.P. Singh; The Bleaching of Pulp, TAPPI Press, Atlanta, USA, is cited.

During pulp bleaching, chlorine-containing bleaching agents produce to different extents chlorinated products which are hardly degradable and for the most part contaminate waste water. Therefore, these processes give rise to environmental concerns, and the use of chlorine-containing bleaching agents, particularly elemental chlorine, is increasingly restrained. This however implies also a loss of bleaching quality, since elemental chlorine has a high bleaching capacity.

Among the chlorine compounds, chlorine dioxide has a better bleaching capacity than hypochlorite, but it is more expensive than hypochlorite, which is more frequently used.

In the state of the art, bleaching processes which do not involve any chlorine compounds are also employed. For these processes, combinations of oxygen/peroxide and ozone respectively are used. The pulp thus bleached is referred to as TCF (total chlorine-free) pulp, since it is bleached using neither elemental chlorine nor chlorine compounds. On the other hand, in the literature a pulp which is not bleached using elemental chlorine but chlorine compounds is referred to as ECF (elemental chlorine-free) pulp.

For the purposes of this specification, an ECF pulp bleached using hypochiorite is referred to as ECF hypochiorite pulp.

In the state of the art, the so-called brightness is a measure of the intensity of bleaching. It is known from the viscose process that there is a relation between the brightness of a bleached pulp and the brightness of the cellulose products produced from this pulp in such a way that pulps having a higher brightness usually may be processed into fibres having also a higher brightness.

It is the object of an aspect of the invention to provide a process whereby cellulose moulded bodies may be produced which have a higher brightness than cellulose moulded bodies produced from an ECF hypochiorite pulp, while the pulps used have the same starting brightness and which otherwise the same procedure is employed.

In accordance with one aspect of the present invention there is provided a process for the production of cellulose fibres exhibiting high brightness, said process comprises the steps of

- bleaching pulp using a totally chlorine-free bleaching (TCF) compound,
- dissolving the pulp bleached using the bleaching compound in an aqueous tertiary amine-oxide, a

mouldable cellulose solution being obtained, and

- processing said mouldable cellulose solution into fibres.

In accordance with another aspect of the present invention there is provided use of a pulp bleached using a totally chlorine-free (TCF) compound for the production of cellulose moulded bodies according to the amine-oxide process.

By means of the process according to the invention, it is possible to particularly produce cellulose fibres having a high brightness, as well as cellulose films according to the dry/wet-spinning process.

It is surprising to those skilled in the art that TCF pulps achieve higher fibre brightnesses than ECF hypochlorite pulps when the starting pulps have the same brightness, with the positive side-effect of being able to produce fibres in a particularly environmentally friendly way.

The invention is also concerned with the bleaching of fibres produced according to the amine-oxide process using chlorine-free bleaching agents such as oxygen, ozone and particularly hydrogen peroxide.

A preferred embodiment of the process according to the invention is characterized in that the fibres or films produced according to the invention are bleached using a bleaching agent, provided that the bleaching agent does not contain chlorine or any chlorine compound.

As the tertiary amine-oxide, N-methylmorpholine-N-oxide has shown particularly good results.

The invention is also concerned with cellulose moulded bodies, particularly cellulose fibres and films, having a high brightness, produceable according to the process according to the invention.

Moreover, the invention is concerned with the use of a pulp bleached using a chlorine-free bleaching compound for the production of cellulose moulded bodies according to the amine-oxide process.

By means of the following Example and Comparative Example, the invention will be explained in more detail.

Example

To produce a TCF pulp, pulp (of the beech sulphite pulp type) first was bleached by means of an alkaline oxygen extraction reinforced by peroxide, afterwards by means of ozone and finally by means of peroxide, according to known processes. These processes are described for instance in R.P. Singh; The Bleaching of Pulp, TAPPI Press, Atlanta, USA, as well as in EP-A - 0 426 652.

The brightness of the TCF pulp obtained, determined according to ISO 3688, was 90.6. The pulp had a rapid cuprammonium viscosity according to Zellcheming ZM IV/30/62 of 19.8 Pa.s, as well as an α content of 90,9.

Thereafter, this TCF pulp was processed in a known way in a stirring vessel into a dope having a composition of 12% of cellulose, 77% of NMMO and 11% of water. The viscosity of the dope was 1630 Pa.s (temp.: 90°C, shearing rate: 0.1 s^{-1}).

The dope was spun into fibres at 120°C through a spinneret having spinning holes exhibiting diameters of 100 μm, according to the known wet/dry-spinning process (see e.g. EP-A - 0 584 318). The brightness of the fibres produced according to the invention was approximately 50.

The CIELAB fibre brightness was determined according to the following method: On the fibres, the colour coordinates R_X, R_Y, R_Z were determined according to DIN 6174 and DIN 5033 using the light type D65 and at a 10° observation angle. From these colour coordinates, according to DIN 55981 (= chromaticity deviation according to Gärtner/Griesser), the CIE brightness and the tint in the red/green-axis may be calculated as follows:

CIE brightness : W = Y + 800* (x0-x) + 1700(y0-y)
Tint : T = 900* (x0-x) -650* (y0-y)
(Note: T negative = red cast, T positive = green cast)
Y... standard chromaticity for green sensitivity (= Ry...reflectance value green)
x0, y0... standard chromaticity coordinates of the achromatic point (at D65/10° is: x0 = 0.3138; y0 = 0.3310)
x, y... standard chromaticity coordinates of the samples; to be calculated according to:
x = X/(X + Y + Z)
y = Y/(X + Y + Z)
z = 1 - x - y
The standard chromaticity numbers X, Y, Z appearing in these formulae may be calculated from the reflectance values (=filter values) Rx, Ry, Rz at the light type D65 and a 10° observation angle according to the formulae:
X = 0.94811 * Rx
Y = Ry
Z = 1.07304 * Rz

Subsequently, these fibres were bleached using hydrogen peroxide (1,5 g of H_2O_2; stabilizing agent: 0,2 g/l of $MgSO_4$; pH 10,5; fleet ratio 1:20; 70°C; bleaching duration: 3 minutes). The CIELAB brightness of the bleached fibres was 57.

Comparative Example

To produce an ECF hypochlorite pulp, first the same starting pulp as in the above Example was bleached by means of an alkaline oxygen extraction reinforced by peroxide, afterwards by means of hypochlorite and finally by means of peroxide.

The brightness of the ECF hypochlorite pulp obtained was 91.6 and thus virtually the same as the one of the TCF pulp produced in the Example.

Afterwards, a dope and fibres were produced analogously to the above Example. The fibres produced had a CIELAB brightness of about 39. Thus it becomes clear that the TCF pulp bleached according to the invention without any chlorine compound may be processed into fibres having a higher brightness than the ECF pulp bleached by means of, among other substances, hypochlorite.

Subsequently, the fibres produced were bleached as described in the above Example. The bleached fibres had a CIELAB brightness of about 54.

CLAIMS:

1. A process for the production of cellulose fibres exhibiting high brightness, said process comprises the steps of
   - bleaching pulp using a totally chlorine-free (TCF) bleaching compound,
   - dissolving said pulp bleached using said bleaching compound in an aqueous tertiary amine-oxide, a mouldable cellulose solution being obtained, and
   - processing said mouldable cellulose solution into fibres.

2. A process according to claim 1, wherein said mouldable cellulose solution is processed into fibres according to the dry/wet-spinning process.

3. A process according to claim 2, wherein said fibres are bleached using the bleaching compound.

4. A process according to one of the claims 1 or 2, wherein as said tertiary amine-oxide N-methylmorpholine-N-oxide is used.

5. Use of a pulp bleached using a totally chlorine-free (TCF) bleaching compound for the production of cellulose moulded bodies according to the amine-oxide process.