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(54) ONE STAGE METHOD FOR ACID METAL REMOVAL AND BLEACH

(57) There is provided a process for chemically pretreating reclaimed cellulose fibres to be used in the production of moulded bodies from regenerated cellulose, wherein the pretreatment includes one stage, in which stage acid metal removal and acid oxidative bleaching are carried out together. Advantages include that the propensity of the regenerated cellulose to clog when flowing in a tube and through a nozzle is reduced. This is believed to be an effect of an efficient metal removal. The need for additional bleaching steps and/or metal removing steps is reduced or even eliminated. A one-stage method is more efficient, faster and less costly compared to a multi-stage method according to the prior art. From an environmental perspective, acidic metal removal is preferred over removal by chelating agents such as EDTA.

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Description

Technical Field

[0001] The invention relates to a one-stage step in a process for regenerating reclaimed cellulose, where acid metal removal is combined together with acid oxidative bleach in one single stage.

Background

[0002] WO 2012/057684 discloses a process for the derivatization of cellulose comprising the sequential steps: a) mixing cellulose with a viscosity below 900 ml/g with an aqueous solution to obtain a liquid, wherein particles comprising cellulose in said liquid have a diameter of maximum 200 nm, wherein the temperature of the aqueous solution is below 20°C, and wherein the pH of the aqueous solution is above 12, b) subjecting the liquid to at least one of the steps: i) decreasing the pH of the liquid with at least 1 pH unit, ii) increasing the temperature by at least 20°C, and c) derivatization of the cellulose.

[0003] WO 2010/124944 discloses a process for the hydrolysis of cellulose comprising the sequential steps (a) mixing cellulose with a viscosity below 900 ml/g with an aqueous solution to obtain a liquid, wherein particles comprising cellulose in said liquid have a diameter of maximum 200 nm, wherein the temperature of the aqueous solution is below 35°C, and wherein the pH of the aqueous solution is above 12, (b) subjecting the liquid to at least one of the steps: (i) decreasing the pH of the liquid with at least 1 pH unit and (ii) increasing the temperature by at least 20°C, and (c) hydrolysing the cellulose. Moreover, there is disclosed glucose manufactured according to the method and ethanol manufactured from the glucose.

[0004] WO 2013/124265 discloses a method for regeneration of a cellulose containing material, comprises the steps: a) exposing the cellulose containing material to oxygen with an alkali aqueous solution at a pH of at least 9, and a temperature of at least 20°C, b) dispersing the cellulose containing material in the alkali aqueous solution, wherein the temperature of the alkali aqueous solution is lowered below 15°C, and wherein the pH of the alkali aqueous solution is above 9, c) adding an organic solvent to the dispersion to precipitate cellulose, and d) separating the precipitated cellulose by at least one method selected from filtering and centrifugation. The method makes it possible to maintain a high alkali pH value in the process, which saves costs since the pH value does not have to be lowered by additions of various additives.

[0005] WO 2018/104330 discloses a cellulose based fibre made of i) a cellulose dissolving pulp, and ii) a recycled cellulose textile, which is treated to swell the cellulose with a reducing additive and a) bleached with oxygen at alkaline conditions with a pH in the range 9-13.5 and/or b) bleached with ozone at acid conditions below pH 6, wherein the cellulose based fibre is manufactured with one selected from a Viscose process and a Lyocell process.

[0006] WO 2018/073177 discloses method for recycling textiles comprising cellulose with the following steps of: optionally disintegrating the textile, swelling the cellulose, under reducing conditions, wherein at least one reducing agent is present at least during a part of the swelling, and then performing at least one of the following two bleaching steps in any order: i) bleaching the material with oxygen at alkaline conditions with a pH in the range 9-13.5, and ii) bleaching the material with ozone at acid conditions below pH 6.

[0007] WO 2015/077807 discloses a process for pretreating reclaimed cellulose fibres to be used in the production of moulded bodies from regenerated cellulose by the Viscose or Lyocell process, wherein the treatment of the reclaimed cellulose fibres includes a chemical metal removing stage and an oxidative bleaching stage. The process is described as a multistage process. The metal removing stage can be acidic washing or treatment with a complexing agent, or a combination of both. The oxidative bleaching stage can be treatment with peroxide, oxygen or ozone.

[0008] Problems in the state of the art include how to further reduce the metal content of regenerated cellulose. Another problem is how to further reduce the clogging properties of regenerated cellulose. In general, it is also desirable to provide a simpler and more efficient method to regenerate cellulose.

Summary

[0009] It is an object of the present invention to alleviate at least some of the problems in the prior art and to provide a method for pre-treating reclaimed cellulose fibres.

[0010] In a first aspect there is provided a process for chemically pretreating reclaimed cellulose fibres to be used in the production of moulded bodies from regenerated cellulose, wherein the pretreatment includes one stage, in which stage acid metal removal and acid oxidative bleaching are carried out together.

[0011] The propensity of the regenerated cellulose to clog when flowing in a tube and through a nozzle is reduced. This is believed to be an effect of an efficient metal removal.

[0012] The need for additional bleaching steps and/or additional metal removing steps is reduced or even eliminated.

[0013] A one-stage method is more efficient, faster and less costly compared to a multi-stage method according to the prior art.

[0014] The invention makes it possible to remove metals efficiently without using complex formers such as EDTA, which may be an environmental issue due to the longevity of EDTA and similar compounds.

Detailed description

[0015] The following detailed description discloses by way of examples details and embodiments by which the invention may be practised.

[0016] It is to be understood that the terminology employed herein is used for the purpose of describing particular embodiments only and is not intended to be limiting since the scope of the present invention is limited only by the appended claims and equivalents thereof.

[0017] If nothing else is defined, any terms and scientific terminology used herein are intended to have the meanings commonly understood by those of skill in the art to which this invention pertains.

[0018] In a first aspect there is provided a process for chemically pretreating reclaimed cellulose fibres to be used in the production of moulded bodies from regenerated cellulose, wherein the pretreatment includes one stage, in which stage acid metal removal and acid oxidative bleaching are carried out together.

[0019] As known in the prior art reclaimed cellulose can be regenerated and used for production of moulded bodies. Examples of such processes include but are not limited to: the Lyocell process, in particular using aqueous amine oxide, such as 4-methylmopholine N-oxide (EP 0356419 and EP 0584318), the viscose process (Kurt Götze, Chemiefasern nach dem Viskoseverfahren, 1967) and the Modal process (AT 287905).

[0020] It has been discovered that by using acid metal removal and acid oxidative bleaching together in one single stage, several advantages can be achieved compared to the prior art. As shown in the appended experimental results the acid metal removal is more efficient compared to removal by complex formation such as addition of EDTA. Further, it can be seen from the experiments that the clogging is reduced for cellulose treated according to the invention.

[0021] Without wishing to be bound by any specific scientific theory the inventors believe that the removal of metal ions reduces the propensity of the cellulose chains to form clusters and cause clogging and other problems. It is believed that in particular multivalent ions such as divalent and trivalent ions, such as Ca^{2+} contribute to the formation of clusters due to their strong electrostatic interactions. For instance, carboxylated cellulose nanofibrils can form hydrogels in the presence of divalent and trivalent ions and form interconnected porous nanofibril networks. When the metal ions are removed, the clogging is reduced as measured in the appended experiments.

[0022] Compared to a multistep method such as the one described in WO 2015/077807, several advantages are achieved. First one single stage is less costly, faster, and more efficient than two or more stages. Secondly, the clogging propensity of the regenerated cellulose is reduced to a greater extent as shown by the appended experimental data.

[0023] Metal ions are present in recycled cellulose and it is desired to reduce the content of such metal ions.

[0024] The stage is one single stage and is intended to be used as a stage in the regeneration of reclaimed cellulose, including recycled clothing, for instance clothing comprising cotton.

[0025] It is conceived that the present stage according to the invention is to be carried out together with additional steps and stages in the regeneration of reclaimed cellulose. Additional stages are known in the art and can easily be combined with the stage according to the invention by a skilled person. A number of additional stages are suitably performed in the regeneration of reclaimed cellulose as described in the prior art. Buttons, zips and other objects of solid metal are in one embodiment removed before the stage. A mechanical treatment is in one embodiment, carried out before the present stage, such as for instance shredding and milling. A chemical treatment step is in one embodiment carried out before the stage according to the invention. Such a chemical treatment step may include a partial or entire dissolution of the reclaimed cellulose.

[0026] In one embodiment, a step of removing non-cellulosic fibres is performed. Examples of such non-cellulosic include but are not limited to fibres comprising polyester, elastan, acryl. In one embodiment, the removal of non-cellulosic fibres is carried out by flotation.

[0027] Any suitable acid can be used in order to obtain an acidic pH during the stage, i.e. a pH value below 7. In order to obtain appropriate pH, mixtures of acids can be used. In one embodiment, a carboxylic acid is present during the stage. The wording that an acid is present during the stage means that it may be present during at least a part of the stage, for instance an acid may be present at the beginning of the stage in order to create the desired pH value and during the stage one or more additional acids can be added. In one embodiment, at least one or more acids are present during a part of the stage. In an embodiment where an acid is present during a part of the stage, it should be ensured that the pH value is acidic as desired during the entire stage, for instance by presence of acid(s) also in the beginning of the stage. Examples of acids which may be present as only acids or together with other acids include but are not limited to hydrochloric acid, formic acid, citric acid, acetic acid. Mixtures including one or more of these acids are also encompassed. Mixture including these acids together with additional acids are also encompassed. In one embodiment, at least one acid selected from the group consisting of hydrochloric acid, formic acid, citric acid, acetic acid and any mixture thereof, is present during the stage. In one embodiment, a mixture of acids is used. For instance, hydrochloric acid can be mixed with a weaker acid such as acetic acid in order to achieve the desired pH. The fact that at least one acid is present during the stage means that the acid can be added in the beginning of the stage, or before the stage or a combination thereof. In one embodiment, the acid is added in the beginning of the stage. Addition of an acid in the beginning of the stage or before the stage does not exclude the possibility of addition of further acid during the stage.

[0028] In one embodiment, the pH is in the interval 1-3 during the stage. In another embodiment the pH is in the interval 2-3 during the stage. In an alternative embodiment, the pH is in the interval 0-4 during the stage. In yet another embodiment the pH is in the interval 1-5 during the stage. In a further embodiment, the pH is in the interval 1.5-5 during the stage. In a still further embodiment, the pH is in the interval 0-5 during the stage.

[0029] In one embodiment, the temperature during the stage is in the interval 40-60 °C. In another embodiment, the temperature during the stage is in the interval 30-75 °C. The temperature during the stage can vary. For instance, the temperature can be high and in the upper part of the interval in the beginning of the stage and lower towards the end. Also a lower initial temperature followed by a rise in the temperature is conceivable. It is not necessary that the temperature is within the ranges during the entire stage, temperatures below and above the ranges are also conceivable. In alternative embodiments, the temperature is outside the ranges during the entire stage.

[0030] In one embodiment, at least one weak acid with a pKa above 3 and below 7 is present during the stage. $3 < pKa < 7$. A weak acid has the advantage that the cellulose is not hydrolysed by the acid, or at least not hydrolysed to any significant degree.

[0031] In one embodiment, acetic acid is present during the stage. Without wishing to be bound by any particular scientific theory, the inventors believe that acetic acid is beneficial for removing dye. Although the mechanisms behind this are not fully understood, the inventors believe that addition of acetic acid gives an improvement in the removal of certain dyes. The stage according to the present invention can suitably be combined with additional bleaching stages in order to fully remove any remaining dyes. Such additional bleaching stages are known and described in the prior art.

[0032] In one embodiment, sulphuric acid is not present during the stage. A common impurity in reclaimed cellulose such as reclaimed cotton is calcium. When sulphuric acid is utilized, Ca^{2+} ions can react with the sulphuric acid and form $CaSO_4$ (gypsum), which makes the process less efficient. It is still possible to use sulphuric acid as evidenced by the examples, but longer treatment time and/or a higher concentration of sulphuric acid would be necessary. Further less efficient metal removal can be expected. Thus the use of sulphuric acid is often less preferred. However, in an alternative embodiment sulphuric acid is present during the stage. If used, sulphuric acid should be used at higher concentration and/or with longer treatment time.

[0033] During the acidic metal removal stage, an acidic oxidative bleach takes place simultaneously in the same stage in order to improve the efficiency. In one embodiment, hydrogen peroxide is present during the stage. The dose of hydrogen peroxide is in one embodiment 2 to 40 kg hydrogen peroxide per odtp. In one embodiment, ozone is present during the stage. In one embodiment, the dose of ozone is 0.1 to 6 kg ozone per odtp. Since the acidic metal removal and the acidic oxidative bleach are carried out together in the same stage, the pH is acidic for all conceivable oxidative bleaching additives.

[0034] The oxidative bleaching agent is added before the step starts or at the start of the stage. In one embodiment, an oxidative bleaching agent is added in the beginning of the stage and at least one oxidative bleaching agent is added during the stage. In one embodiment, at least one oxidative bleaching agent is added in the beginning of the stage and at least one additional different oxidative bleaching agent is added during the stage. In one embodiment, ozone is added in the beginning of the stage and hydrogen peroxide is added during the stage at a later point in time. In another embodiment hydrogen peroxide is added in the beginning of the stage and ozone is added during the stage. Also other combinations of additions of oxidative bleaching agents are encompassed.

[0035] In one embodiment, the stage with both acid metal removal and acid oxidative bleaching is the only acidic chemical metal removal step in the process. Even if the detailed mechanisms are not fully investigated the inventors believe that the acidic metal removal and the acidic oxidative bleach should be carried out together in one stage. If acidic metal removal and the bleach are carried out separately the efficiency is lower as seen in the examples. Thus the combined acid metal removal and acid oxidative bleaching is in one embodiment the only chemical metal removal stage in the process, i.e. no additional acid chemical metal removal stages are carried out before or after the stage according to the invention. The metal removal according to the invention is so efficient that it is more economical to use only one efficient stage and no additional acidic metal removal stages. In one embodiment, the acidic metal removal in the stage is the only acidic metal removal.

[0036] In one embodiment, the stage is carried out during a time in the interval from 1 to 60 minutes. In yet another embodiment the stage is carried out during 2-60 minutes. In yet another embodiment the stage is carried out during 5-60 minutes. In yet another embodiment the stage is carried out during 10-60 minutes. Longer treatment times can also be used, but can also be less economical. Thus In one embodiment, the stage is carried out during a time of at least 1 minute, at least 2 minutes, at least 5 minutes, or at least 10 minutes.

[0037] The reclaimed cellulose fibres are in one embodiment, cotton fibres. In one embodiment, the cellulose fibres originate from pre-consumer cellulose containing waste such as cotton. Pre-consumer cellulose containing waste includes but is not limited to combing waste and cuttings. In one embodiment, the cellulose fibres originate from post-consumer cellulose containing waste such as cotton. Post-consumer cellulose containing waste includes but is not limited to laundry waste and used clothes. In one embodiment, the cellulose fibres comprise pulp prepared from cotton rags. In one embodiment, the reclaimed cellulose is mechanically shredded or milled prior to the use.

[0038] In one embodiment, the production of moulded bodies is made with the Viscose process. In one embodiment, the production of moulded bodies is made with the Lyocell process. In one embodiment, the production of moulded bodies is made with the Modal process. These processes for production of moulded bodies are known in the art and can be performed by a skilled person. The intrinsic viscosity of the regenerated cellulose is suitably adjusted as needed and as known depending on the intended production process for the moulded bodies. For instance, for a Viscose process the intrinsic viscosity can be adjusted to a value in the range 350-650 ml/g. For instance, for a Lyocell process the intrinsic viscosity can be adjusted to 350-500 ml/g.

Examples

[0039] In order to show the advantageous properties of the present invention a clogging test was performed. A mixture to be tested was allowed to run through a narrow passage. The times for the passing of 25-50 ml was recorded as well as the time for the passing of 125-150 ml. The difference was recorded as delta T (Δt).

Example 1

[0040] Reactivity test based on wash, white jersey fabric. The samples were treated mechanically by cutting in pieces 1x1 cm and thereafter treated in a mixer for 40 seconds. This shredding served to open the fiber structure. After the mechanical step, the material was treated in a chemical step where the material was bleached at high pH, i.e. a pH above 7.

[0041] The following three samples were prepared:

	H_2O (1)	EDTA (2)	H_2SO_4 (3)
Mass fabric(g, od)	50	50	50
EDTA (g/mL)	N/A	0.3	N/A
H_2SO_4 (mol/L)	N/A	N/A	0.01 M (pH 2)

[0042] Samples with a weight of 3.75 g were treated with ozone during 10 min with an amount corresponding to 238 kg ozone/hour. Ozone treated pulp samples were then wetted overnight and diluted to a concentration of 3.5 wt%. The initial ozone treatment was a separate oxidative bleaching step. Thereafter each pulp sample was washed with the additive according to the above table.

- H_2O (1): was washed with 1.5 1 deionized water.
- EDTA (2): was washed with EDTA-solution (2 liters, 60 °C) and thereafter with 1.5 1 deionized water.
- H_2SO_4 (3) : was washed with H_2SO_4 (2 liters, 0.01 M, 60 °C) followed by 1.5 1 deionized water.

[0043] The second wash was a different metal removal step.

[0044] After the different washes the samples were diluted to a concentration of 9 wt% and the pH was adjusted to 6 for all samples with NaOH and if needed with H_2SO_4.

[0045] Delta T (Δt) was measured for the samples and the results are shown in the below table.

Results

[0046]

Sample	**t1 (25-50 ml) [s]**	**t2 (125-150 ml) [s]**	**Δt [s]**
H_2O (1)	55	215	160

(continued)

Sample	t1 (25-50 ml) [s]	t2 (125-150 ml) [s]	Δt [s]
EDTA (2)	24	53	29
H_2SO_4 (3)	51	177	126

[0047] As can be seen the acidic metal removal is not very efficient compared to the control sample with water. There is still a considerable clogging, which is attributed to metal ions present in the regenerated cellulose. Metal removal with the chelating agent ethylenediaminetetraacetic acid (EDTA) is more efficient compared to sulphuric acid. However, environmental safety has raised concerns about the low biodegradability of aminopolycarboxylates such as EDTA.

Example 2

[0048] Reactivity test based on wash, white jersey fabric. First, the material was subjected to a mechanical step and thereafter in a chemical step, both as in example 1. Thereafter the following three samples were prepared.

	(1) H_2SO_4	(2) HCl	(3) Acetic acid
Mass fabric(g, od)	50	50	50
H_2O_2 (kg/ton)	2	2	2

[0049] The samples were wetted overnight and diluted to a concentration of 3.5 wt%. Thereafter the pH value of each pulp sample was adjusted and H_2O_2 was added.

- H_2SO_4: pH was adjusted to 2 with H_2SO_4.
- HCl: pH was adjusted to 2 with HCl.
- Acetic acid: pH was adjusted to 2.4 with acetic acid.

[0050] After the pH adjustment, the samples had a dwell time of about 20 minutes before wash with deionized water. This gave a stage with combined acidic metal removal and acidic oxidative bleach. All samples were washed with 1.5 liters deionized water after filtering.

[0051] After the washing, the samples were diluted to a concentration of 9 wt% and the pH was adjusted to 6 for all samples with NaOH. If needed the pH was also adjusted with H_2SO_4 for the H_2SO_4-sample and with HCl for the HCl-sample and the acetic acid-sample.

[0052] Delta T (Δt) was measured for the samples and the results are shown in the below table.

Results

[0053]

Sample	t1 (25-50 ml) [s]	t2 (125-150 ml) [s]	Δt [s]
H_2SO_4	17.6	26.3	8.7
HCl	7.5	12.6	5.1
HAc	8.8	12.5	3.7

[0054] As can be seen there is a noticeable improvement in the Δt by using the treatment according to the invention, in particular when not using H_2SO_4.

Claims

1. A process for chemically pretreating reclaimed cellulose fibres to be used in the production of moulded bodies from regenerated cellulose, wherein the pretreatment includes one stage, in which stage acid metal removal and acid oxidative bleaching are carried out together.

2. The process according to claim 1, wherein at least one acid selected from the group consisting of hydrochloric acid, formic acid, citric acid, acetic acid and any mixture thereof, is present during the stage.

3. The process according to any one of claims 1-2, wherein at least one weak acid with a pKa above 3 and below 7 is present during the stage.

4. The process according to any one of claims 1-3, wherein acetic acid is present during the stage.

5. The process according to any one of claims 1-4, wherein sulphuric acid is not present during the stage.

6. The process according to any one of claims 1-4, wherein sulphuric acid is present during the stage.

7. The process according to any one of claims 1-6, wherein hydrogen peroxide is present during the stage.

8. The process according to any one of claims 1-7, wherein ozone is present during the stage.

9. The process according to any one of claims 1-8, wherein the stage with both acid metal removal and acid oxidative bleaching is the only acid chemical metal removal step in the process.

10. The process according to any one of claims 1-9, wherein the stage is carried out during a time in the interval from 1 to 60 minutes.

11. The process according to any one of claims 1-10, wherein the temperature during the stage is in the interval 30-75 °C.

12. The process according to any one of claims 1-11, wherein a subsequent production of moulded bodies from regenerated cellulose is carried out with one method selected from the group consisting of the Viscose process, the Lyocell process, and the Modal process.

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EUROPEAN SEARCH REPORT

Application Number
EP 20 16 3367

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D01F
D01D
D06M
D06L
D21H
D21C

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
The Hague	18 August 2020	Fiocco, Marco

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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