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(71) Applicant: Zhejiang Jingxing Paper Joint Stock Co., Ltd.

314214 Pinghu City, Zhejiang (CN)

(72) Inventors:

 Cao, Haibing Pinghu City, Zhejiang, 314214 (CN)

- An, Xingye
 Pinghu City, Zhejiang, 314214 (CN)
- Lu, Bin Pinghu City, Zhejiang, 314214 (CN)
- Liu, Hongbin Pinghu City, Zhejiang, 314214 (CN)
- Zhang, Runqing Pinghu City, Zhejiang, 314214 (CN)
- Xu, Dong Pinghu City, Zhejiang, 314214 (CN)
- Dong, Hongming Pinghu City, Zhejiang, 314214 (CN)
- (74) Representative: 2s-ip Schramm Schneider Bertagnoll
 Patent- und Rechtsanwälte Part mbB
 Postfach 86 02 67
 81629 München (DE)

(54) METHOD FOR IMPROVING FIBER SOFTNESS OF HIGH YIELD PULP

Disclosed is a method for improving fiber softness of high yield pulp, including a high yield pulp; the method comprises: treating the high yield pulp with ozone; and treating with cellulase; the disclosure offers the following advantages: The high yield pulp in the present disclosure is sequentially treated with ozone and cellulase. Removal of lignin on the surface with ozone increases the softness of the fiber and loosens the fiber structure. Therefore, treating the fiber with ozone can open a "channel" for the entry of cellulase. Then the cellulase allows water molecules to enter the fiber, and the distance between fiber macromolecule chains increases. which causes the fiber to deform with reduced stiffness, thereby improving the softness. Therefore, by treating with cellulase based on the pretreatment of high-yield pulp with ozone, the present disclosure increases the accessibility of cellulase and fiber. Besides, ozone as a green agent can improve the softness of the fiber without or with little pollution to the environment.

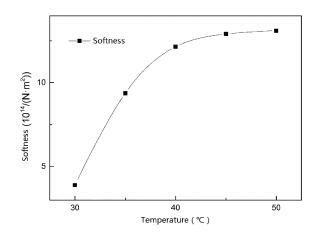


FIG. 1

Description

FIELD

[0001] The present disclosure relates to a method for improving fiber softness of high yield pulp.

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BACKGROUND

[0002] As a pulping technology with high yield, high quality, and low pollution, the chemimechanical pulping process emerged in 1970s, which is one kind of high yield pulping. The high yield pulp obtained from high yield pulping has characteristics of high yield and low pollution, such that a paper factory can manufacture products with premium quality at a lower cost. It can be said that high yield pulp fills up the blank between conventional mechanical pulp and chemical pulp in respect of performance. In recent years, owing to its so many functional advantages in papermaking, such as high bulk factor, high stiffness, large opacity, good dimensional stability, and good printability, etc. the high yield pulp has gained wide applications in various kinds of paper and paper boards.

[0003] With improvement of quality of life, people focus more and more on paper softness, which gradually exposes the drawback of high yield pulp, i.e. poor softness, one of bottlenecks restricting development of the high yield pulp. The Chinese patent No. 2016105224934 discloses a method of manufacturing a paper towel containing high yield pulp, comprising: subjecting the high yield pulp to refiner beating through a crushing-type beating process, wherein the beating consistency is 20%, and the freeness of the pulp is controlled to 250~400ml. On the one hand, pretreating the high yield pulp using the crushing-type beating process will reduce the volume of fiber cavities, thereby affecting the water retention value of pulp fibers and water-absorptivity of paper towels; and on the other hand, the crushing pretreatment has less impact on lignin of the fiber, thereby affecting inter-fiber bonding force in the paper towel and reducing the mechanical strength of the paper towel.

SUMMARY

[0004] An objective of the present disclosure is to provide a method for improving fiber softness of high yield pulp, which enables removal of part of lignin from the high yield pulp fiber to thereby improve fiber softness.

[0005] To achieve the objective above, the present disclosure provides the following technical solution: a method for improving fiber softness of high yield pulp, including a high yield pulp with a pulp consistency of 1~3%, the method comprising steps of:

[0006] Step 1: treating the high yield pulp with ozone, wherein the concentration of ozone is $10\sim30\%$, the treatment temperature is $30\sim50$ °C, the treatment duration is $60\sim210$ s, and the pH value during treatment is $2.5\sim4.5$;

[0007] Step 2: treating the treated high yield pulp from step 1 with cellulase, wherein the content of cellulase is 0.1~3EGU/g, the treatment duration is 30~60min, the PH value during treatment is 45~60°C.

[0008] Preferably, the treatment temperature in step 1 is 30°C, 35°C, 40°C, 45°C or 50°C, respectively; the concentration of ozone is 10%; the treatment duration is 120s, the PH value during treatment is 3, and the consistency of the high yield pulp is 2%.

[0009] Preferably, the consistency of high yield pulp in step 1 is 1.5%, 2%, 2.5%, 3% or 3.5%, respectively; the treatment temperature is 30°C, the concentration of ozone is 10%; the treatment duration is 120s, the pH value during treatment is 3.

5 [0010] Preferably, the temperature when treating the high yield pulp with ozone is controlled and adjusted by water bath.

[0011] Preferably, the ozone reacts with the high yield pulp in a three-neck flask.

[0012] Preferably, the cellulase reacts with the high yield pulp in a 1000ml beaker.

[0013] Preferably, the cellulase in step 2 includes one of or a mixture of at least two of endoglucanase, exoglucanase, and β -glycosidase.

[0014] In view of the above, the present disclosure offers the following advantages: the high yield pulp in the present disclosure is sequentially treated with ozone and cellulase. Because the ozone has a relatively low dissolvability, it can hardly enter the inside of the fiber; therefore, it first acts on the fiber surface such that ozonation attacks the primary wall lignin, the outer wall of the secondary wall and the intercellular lignin, wherein the lignin side chain is oxidized (by polymer depolymerization), the aromatic ring is broken (ring-opened), forming an organic acid which is dissolved in water. With loss of yield, the primary wall and the intercellular layer selectively remove lignin and expose the surface hydrophilic substances. The removal of lignin on the surface increases the softness of the fiber and loosens the fiber structure. Therefore, treating the fiber with ozone can open a "channel" for the entry of cellulase. Then the cellulase can hydrolyze the amorphous region of fiber, allowing water molecules to enter the fiber, and the distance between fiber macromolecule chains increases, which causes the fiber to deform with reduced stiffness, thereby improving the softness. Therefore, by treating with cellulase based on the pretreatment of high-yield pulp with ozone, the present disclosure increases the accessibility of cellulase and fiber. With an intention to reduce use of cellulase and improve the post-treatment fiber softness, the present disclosure offers a dual-beneficial and prospective process for treating high yield pulp; besides, ozone as a green agent can improve the softness of the fiber without or with little pollution to the environment.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015] Hereinafter, the present disclosure will be fur-

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ther illustrated with reference to the accompanying drawings:

Fig. 1 shows a schematic diagram of softness with ozone under different temperatures;

Fig. 2 shows a schematic diagram of softness with ozone under different pulp consistencies.

DETAILED DESCRIPTION OF EMBODIMENTS

[0016] A method for improving fiber softness of high yield pulp, including a high yield pulp with a pulp consistency of $1\sim3\%$, the method comprising steps of:

[0017] Step 1: treating the high yield pulp with ozone, wherein the concentration of ozone is $10\sim30\%$, the treatment temperature is $30\sim50^{\circ}$ C, the treatment duration is $60\sim210$ s, and the PH value during treatment is $2.5\sim4.5$; [0018] Step 2: treating the treated high yield pulp from step 1 with cellulase, wherein the content of cellulase is $0.1\sim3$ EGU/g, the treatment duration is $30\sim60$ min, the PH value during treatment is $45\sim60^{\circ}$ C.

[0019] The high yield pulp in the present disclosure is sequentially treated with ozone and cellulase. Because the ozone has a relatively low dissolvability, it can hardly enter the inside of the fiber; therefore, it first acts on the fiber surface such that ozonation attacks the primary wall lignin, the outer wall of the secondary wall and the intercellular lignin, wherein the lignin side chain is oxidized (by polymer depolymerization), the aromatic ring is broken (ring-opened), forming an organic acid which is dissolved in water. With loss of yield, the primary wall and the intercellular layer selectively remove lignin and expose the surface hydrophilic substances. The removal of lignin on the surface increases the softness of the fiber and loosens the fiber structure. Therefore, treating the fiber with ozone can open a "channel" for the entry of cellulase. Then the cellulase can hydrolyze the amorphous region of fiber, allowing water molecules to enter the fiber, and the distance between fiber macromolecule chains increases, which causes the fiber to deform with reduced stiffness, thereby improving the softness. Therefore, by treating with cellulase based on the pretreatment of high-yield pulp with ozone, the present disclosure increases the accessibility of cellulase and fiber. With an intention to reduce use of cellulase and improve the posttreatment fiber softness, the present disclosure offers a dual-beneficial and prospective process for treating high yield pulp; besides, ozone as a green agent can improve the softness of the fiber without or with little pollution to the environment.

[0020] The temperature for treating the high-yield pulp with ozone is controlled via a water bath. The water bath is simple to operate and has a good heating effect, such that it may guarantee that the adjusted temperature reaches the set temperature range to thereby guarantee the thermostatic effect of the temperature. The ozone reacts with the high yield pulp via a three-neck flask,

which is convenient for adjusting ozone concentration and pH value during the test; the adjustment of the ozone concentration can be independent from the adjustment of pH value, thereby improving the efficiency of adjustment. The cellulase reacts with the high yield pulp via a 1000ml beaker, which facilitates stirring during the cellulase treatment to thereby improve the treatment effect; the cellulase in step two includes one of or a mixture of at least two of endoglucanase, exoglucanase, and β -glycosidase, which may be adjusted according to different high-yield pulps so as to meet different needs, thereby offering a good applicability.

First Example:

[0021] A method for improving fiber softness of high yield pulp, including a high yield pulp with a pulp consistency of 2%, the method comprising steps of:

Step 1: treating the high yield pulp with ozone, wherein the treatment temperature was 30°C, 35°C, 40°C, 45°C or 50°C, respectively; the concentration of ozone was 10%; the treatment duration was 120s, the PH value during treatment was 3, and the consistency of the high yield pulp was 2%;

Step 2: treating the treated high yield pulp from step 1 with cellulase, wherein the content of cellulase was 0.5EGU/g, the treatment duration was 30min, the PH value during treatment was 45°C.

[0022] The treated high yield pulp was subjected to softness detection, wherein the curve relationship between the detected softness and the treatment temperatures is shown in Fig. 1.

Second Example:

[0023] A method for improving fiber softness of high yield pulp, including a high yield pulp with a pulp consistency of 2%, the method comprising steps of:

Step 1: treating the high yield pulp with ozone, wherein the treatment temperature was 30°C; the concentration of ozone was 10%; the treatment duration was 120s, the PH value during treatment was 3, and the consistency of the high yield pulp was 1.5%, 2%, 2.5%, 3% or 3.5%, respectively;

Step 2: treating the treated high yield pulp from step 1 with cellulase, wherein the content of cellulase was 0.5EGU/g, the treatment duration was 30min, the pH value during treatment was 45°C.

[0024] The treated high yield pulp was subjected to softness detection, wherein the curve relationship between the detected softness and the consistencies of the high yield pulp is shown in Fig. 2.

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[0025] Additionally, the ozone treatment in step 1 may also treat the high yield pulp by changing the ozone consistency, or the ozone treatment duration, or the $_{\rm p}$ H value during treatment.

[0026] Besides the preferred embodiments above, the present disclosure also has other embodiments. Those skilled in the art may make various variations and alternations based on the present disclosure, and such variations and alterations should fall within the scope defined by the appended claims without departing from the spirit of the present disclosure.

cellulase reacts with the high yield pulp in a 1000ml beaker.

7. The method for improving fiber softness of high yield pulp according to claim 1, wherein the cellulase in step 2 includes one of or a mixture of at least two of endoglucanase, exoglucanase, and β-glycosidase.

Claims

1. A method for improving fiber softness of high yield pulp, including a high yield pulp with a pulp consistency of 1~3%, the method comprising steps of:

Step 1: treating the high yield pulp with ozone, wherein the concentration of ozone is $10\sim30\%$, the treatment temperature is $30\sim50$ °C, the treatment duration is $60\sim210$ s, and the PH value during treatment is $2.5\sim4.5$;

Step 2: treating the treated high yield pulp from step 1 with cellulase, wherein the content of cellulase is $0.1\sim3EGU/g$, the treatment duration is $30\sim60$ min, the PH value during treatment is $45\sim60$ °C.

- 2. The method for improving fiber softness of high yield pulp according to claim 1, wherein the treatment temperature in step 1 is 30°C, 35°C, 40°C, 45°C or 50°C, respectively; the concentration of ozone is 10%; the treatment duration is 120s, the PH value during treatment is 3, and the consistency of the high yield pulp is 2%.
- 3. The method for improving fiber softness of high yield pulp according to claim 1, wherein the consistency of high yield pulp in step 1 is 1.5%, 2%, 2.5%, 3% or 3.5%, respectively; the treatment temperature is 30°C, the concentration of ozone is 10%; the treatment duration is 120s, the PH value during treatment is 3
- 4. The method for improving fiber softness of high yield pulp according to any one of claims 1~3, wherein the temperature when treating the high yield pulp with ozone is controlled and adjusted by water bath.
- 5. The method for improving fiber softness of high yield pulp according to any one of claims 1~3, wherein the ozone reacts with the high yield pulp in a three-neck flask.
- **6.** The method for improving fiber softness of high yield pulp according to any one of claims 1~3, wherein the

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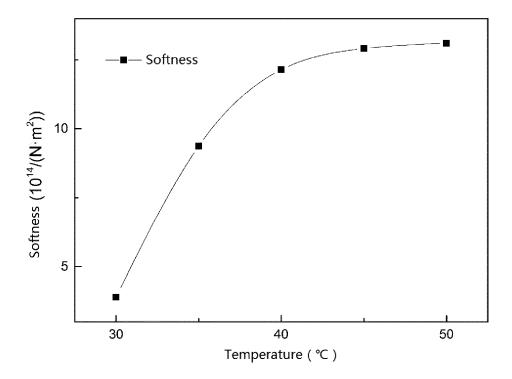
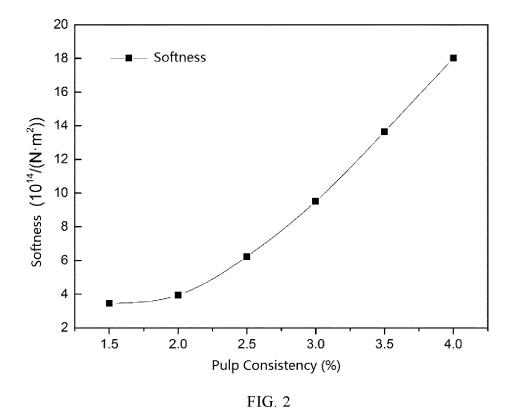


FIG. 1





Category

EUROPEAN SEARCH REPORT

DOCUMENTS CONSIDERED TO BE RELEVANT Citation of document with indication, where appropriate, of relevant passages

Application Number EP 19 21 9097

CLASSIFICATION OF THE APPLICATION (IPC)

Relevant

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X	W0 2014/029909 A1 (27 February 2014 (2 * the whole documen		1-7	INV. D21H21/22 D21C5/00
Α	WO 2013/188657 A1 (TRUSTEES [US]) 19 December 2013 (2 * the whole documen		1-7	D21C9/153 D21H11/10 D21H17/00 D21H21/32
A	US 2006/102299 A1 (AL) 18 May 2006 (20 * claims 1-45 *	ELGARHY YASSIN [CA] ET 06-05-18)	1-7	
Α	WO 96/00811 A1 (SCC 11 January 1996 (19 * the whole documen	96-01-11)	1-7	
Α	CN 108 071 038 A (U TECH) 25 May 2018 (* the whole documen		1-7	
A	WO 2016/080895 A1 (26 May 2016 (2016-0 * the whole documen	5-26)	1-7	TECHNICAL FIELDS SEARCHED (IPC) D21C D21H
A	WO 95/13415 A1 (ECC 18 May 1995 (1995-6 * the whole documen	5-18)	1-7	
	The present search report has l	•		
	Place of search Munich	Date of completion of the search 9 July 2020	Kar	lsson, Lennart
X : parl Y : parl doci A : tech O : nor	ATEGORY OF CITED DOCUMENTS ticularly relevant if taken alone ticularly relevant if combined with anot ument of the same category nnological background n-written disclosure rmediate document	L : document cited fo	ument, but publise the application r other reasons	shed on, or

EP 3 770 322 A1

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 19 21 9097

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

09-07-2020

	ent document in search report		Publication date		Patent family member(s)		Publication date
WO 2	2014029909	A1	27-02-2014	BR CA CN EP FI JP NO PL US WO ZA		A1 A1 B B2 A T3 T3 A1 A1	04-07-2017 27-02-2014 13-05-2015 24-06-2015 15-11-2017 20-06-2018 29-10-2015 22-09-2018 31-08-2018 06-08-2015 27-02-2014 29-06-2016
WO 2	013188657	A1	19-12-2013	BR CA CN EP ES JP PL PT US WO	112014031092 2876082 104583492 2861799 2744788 2015521694 2861799 2861799 2015167243 2013188657	A1 A1 T3 A T3 T	27-06-2017 19-12-2013 29-04-2015 22-04-2015 26-02-2020 30-07-2015 31-01-2020 26-09-2019 18-06-2015 19-12-2013
US 2	2006102299	A1	18-05-2006	CA US	2523736 2006102299		17-05-2006 18-05-2006
	0600811	A1	11-01-1996	AT AU BR CA CN DE EP ES JP KR VP PT RU US WO		B2 A A1 A T2 A1 T3 A B1 A A1 E C1 A	15-08-2002 09-04-1998 02-09-1997 11-01-1996 17-09-1997 10-04-2003 16-04-1997 16-02-2003 16-06-1998 27-08-2004 26-08-1998 23-06-1997 29-11-2002 10-03-1999 10-12-1996 22-02-2000 11-01-1996
CN 1	.08071038	A	25-05-2018	NON	IE		

 $\stackrel{ ext{O}}{ ext{L}}$ For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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EP 3 770 322 A1

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 19 21 9097

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

09-07-2020

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 2016080895	A1	26-05-2016	BR 112017009879 A2 CA 2967571 A1 CL 2017001243 A1 EP 3221511 A1 JP 6474897 B2 JP 2017538044 A SE 1451409 A1 US 2017314197 A1 WO 2016080895 A1	26-12-20 26-05-20 01-12-20 27-09-20 27-02-20 21-12-20 22-05-20 02-11-20 26-05-20
WO 9513415	A1	18-05-1995	AU 8129994 A WO 9513415 A1	29-05-19 18-05-19
for more details about this annex				

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EP 3 770 322 A1

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

• CN 2016105224934 [0003]