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(54) **Method for removal of metals from a bleach plant filtrate system**

(57) A method is disclosed for removal of non process elements from a Kraft bleach plant producing lyocell pulp. In the method the bleach plant filtrate is passed through at least one centrifugal cleaner. The non process

elements are reduced by at least 20 percent of their original levels in the bleach plant filtrate.

EP 1 739 225 A2

Description

[0001] This application relates to the removal of non process elements from a Kraft bleach plant producing lyocell pulp. More particularly it relates to the removal of the non process elements from the bleach plant filtrate stream.

[0002] Transition metals are undesirable in lyocell pulp because they accelerate the degradation of cellulose and NMMO in the lyocell process resulting in a lower degree of depolymerization than desired in cellulose and degradation of the NMMO. Examples of transition metals commonly found in pulp derived from trees and other sources include iron, copper, nickel and manganese. For lyocell pulp manufacture it is desirable that the total level of copper, manganese and iron is less than 10 ppm. Silica and calcium are also deleterious in that they interfere with spinning the fiber causing spinneret restrictions and fiber breakage. Silica levels of less than 50 ppm are desirable for lyocell pulp.

[0003] Typically low metal levels in pulp are achieved in lyocell pulp by washing the pulp in the final stages of bleaching with fresh water. Many dissolving mills use ionexchange water treatment for a portion of the make-up to the final bleach washer and /or dilution water used in the wet-end of a paper machine.

[0004] In some mills the above process cannot be used due to environmental restrictions. In some cases, all of the extracted wash water from the final stage of bleaching in a OD₁E_pD₂ sequence is used to wash the pulp in the first stage of bleaching. In lyocell production, the pH of the extracted wash water from the final bleach stage is reduced to approximately 3.1. A large portion of the metals are washed off the pulp and are dissolved or suspended in the extracted filtrate. When the D₂ filtrate is used to wash the D₁ stage pulp, none of the D₂ filtrate is removed. A large caustic dose is added to the D₁ pulp that leaves the D₁ washer. The metals and silica removed from the D₂ washed pulp form complexes or are substantive to the pulp during the alternating acidic and alkaline conditions. These particles have a higher density than pulp and need to be removed to reduce the metals and other elements in the pulp.

[0005] In one embodiment of the invention, an acidic bleach plant filtrate from a lyocell pulp bleach sequence is processed through a centrifugal cleaner. Each of the non process elements may be reduced by at least 20 % of their original levels in the bleach plant filtrate. As defined herein, non process elements include, but are not limited to manganese, iron, copper, calcium and silica. Typical levels of these elements in the bleach plant filtrate are 3 to 9 ppm manganese, 80 to 170 ppm iron, 6 to 13 ppm copper, 3200 to 5500 ppm calcium and 190 to 370 ppm silica. In another embodiment the manganese is reduced by at least 50 percent of the original level in the bleach plant filtrate. In yet another embodiment manganese is reduced by at least 70 percent of the original level in bleach plant filtrate. The acidic filtrate stream works best since the metal particles are liberated from the pulp fiber. Silica particles tend to contain a significant concentration of transition metals which are removed with the silica. In one embodiment silica may be removed by at least 50 % of the original level in the bleach plant filtrate. In another embodiment silica may be removed by at least 70 % of the original level in the bleach plant filtrate. Metal ions that are fully dissolved are not removed in the process. It may be possible to remove these by addition of chelating agents in the bleach plant or addition of polymeric cationic flocculants such as polymers of polyacrylamide or with anionic flocculants such as polyacrylic acid or ethylenediamine-tetraacetic acid to the bleach plant filtrate stream before processing through the centrifugal cleaners.

[0006] Centrifugal cleaners suitable for removal of non process elements include forward and reverse centrifugal cleaners. These are available from Krebs, Tuscon, AZ; GL&V Sweden AB, Stockholm, Sweden; Noss, Darmstadt, Germany; Andritz, Alpharetta, GA; Kadant Black Clawson, Mason, OH and Metso Paper Norcross, GA. Forward centrifugal cleaners can be used alone or in a series while reverse cleaners can be used with a single pass through the centrifugal cleaner cone. The units can be positioned after any stage in the lyocell pulp bleach plant sequence and can process a filtrate stream in a bleach plant up to 5 % solids content. In one embodiment the centrifugal cleaner is positioned after the last bleaching stage. In another embodiment the centrifugal cleaner is positioned after first bleach stage. In yet another embodiment the centrifugal cleaner is positioned after the second stage. Depending on the number of bleach stages, a centrifugal cleaner can be positioned after each stage. It is understood that either a single centrifugal cleaner or a series of centrifugal cleaners can be positioned after any bleach stage or stages.

[0007] Removal of the non process element manganese through a Noss Radiclone centrifugal cleaner, (a forward cleaner), is shown in Table 1 below; the process conditions are shown in the working example.

Table 1

Manganese Removal In Each Process Stream.					
Process Stream	Total Solid Concentration (%)	Mn Concentration (ppm on solids)	Removal (%)	Total Volumetric Flow Split (%)	Mn Mass Flow Split (%)
Feed	0.0027	6.5	-	n/a	n/a
Accept	0.0009	5.0	74	88	21

EP 1 739 225 A2

(continued)

Manganese Removal In Each Process Stream.					
Process Stream	Total Solid Concentration (%)	Mn Concentration (ppm on solids)	Removal (%)	Total Volumetric Flow Split (%)	Mn Mass Flow Split (%)
Reject	0.009	6.9	26*	12	79
*amount remaining with rejects					

[0008] Solids were measured as follows. A Whatman 47 mm glass microfiber filter (934-AH) was washed with distilled water and dried for at least one hour at 103° C-105° C, weighed and inserted into the filtering apparatus, wetted to seat the filter medium and suction applied. One hundred ml. of the process stream was poured into filter apparatus with suction and washed successively with three 10 ml. volumes of distilled water. The filter was then dried for at least one hour at 105° C, weighed and the solids content calculated. Manganese was determined by EPA Method 6010C (SW-846) using inductively coupled plasma spectrometry. The accept filtrate stream, the stream that results from passing the last bleach stage washing over drum washers and then through the centrifugal cleaner in the example, can be used to wash the pulp from any previous stage. Alternatively, there can be a series of centrifugal cleaners in which the resulting accept stream from the centrifugal cleaners can be processed and ultimately used to wash the pulp from any stage in the bleach sequence. Rejects resulting from the centrifugal cleaner or centrifugal cleaners can go to the sewer or could be used in other areas of the pulp mill outside of the fiberline such as the power boiler sand / solids hopper sluice water, the log wash area or to control woodyard dust.

WORKING EXAMPLE

[0009] Wash filtrate from the second chlorine dioxide stage (D₂) of a lyocell pulp bleach sequence was processed through a Noss Radiclone AM80C centrifugal cleaner (Noss, Darmstadt, Germany) to remove non process elements from the filtrate stream before it was used to wash pulp from the first chlorine dioxide stage (D₁) in a OD₁E_pD₂ sequence. D₂ filtrate was pumped through the Noss Radiclone centrifugal cleaner under the following conditions:

Feed Flow rate 94.6 l/min. (25 gpm); 172.37 kPa (25 psig); Temperature, 68.3 °C, (155 °F)

Reject Flow rate- 11.36 l/min., 3 gpm ; 48.26 kPa (7 psig)

Accept Flow rate- 83.28 l/min. (22 gpm); 82.73 kPa (12 psig)

18.93 liters (five gallon sample) of the feed, accept and rejects streams were collected under the above conditions.

The results of manganese removal are shown in Table 1 above.

EXAMPLE 1

[0010] Wash filtrate from the second chlorine dioxide stage (D₂) of a lyocell pulp bleach sequence is processed through a Noss Radiclone AM80C centrifugal cleaner to remove non process elements from the filtrate stream before it is used to wash pulp from the first chlorine dioxide stage (D₁) in a OD₁E_pD₂ sequence. D₂ filtrate is pumped through the Noss Radiclone centrifugal cleaner under the following conditions:

Feed Flow rate - 94.6 l/min. (25 gpm); 172.37 kPa (25 psig); Temperature, 68.3 °C, 155 °F

Reject Flow rate - 11.36 l/min., (3 gpm) ; 48.26 kPa (7 psig)

Accept Flow rate - 83.28 l/min. (22 gpm); 82.73 kPa (12 psig)

18.93 liters (five gallon sample) of the feed, accept and rejects streams are collected under the above conditions.

It is believed that silica and other non process elements (calcium, copper, iron) may be reduced by at least 20 % of their original levels in the bleach plant filtrate. Silica can be determined by ASTM D4517-04, copper, iron and calcium by EPA Method 6010C (SW-846).

[0011] The embodiments of this invention, including the examples, are exemplary of numerous embodiments that may be made of this invention. It is contemplated that numerous other configurations of the process may be used and the equipment used in the process may be selected from numerous sources other than those specifically disclosed. In short, it is the applicant's intention that the scope of the patent issuing herefrom will be limited only by the scope of the appended claims.

Claims

1. A method comprising the steps of:

5 providing a centrifugal cleaner,
providing an acidified bleach plant filtrate comprising non process elements, passing said filtrate through said cleaner,
whereby each of said non process elements are reduced in the accept stream by at least 20% of their original
10 levels in the bleach plant filtrate.

2. A method as claimed in claim 1, wherein said filtrate is a lyocell pulp bleach plant filtrate.

3. A method as claimed in claim 1 or claim 2, wherein the non process elements are selected from the group consisting
15 of manganese, iron, copper, calcium and silica and mixtures thereof.

4. A method as claimed in claim 3, wherein the element is manganese.

5. A method as claimed in claim 3 or claim 4, wherein the element is iron.

20 6. A method as claimed in any of claims 3 to 5, wherein the element is copper.

7. A method as claimed in any of claims 3 to 6, wherein the element is silica.

8. A method as claimed in any of claims 3 to 7, wherein the element is calcium.

25 9. A method as claimed in any of claims 3 to 8, wherein manganese is reduced by at least 50% of the original level in the bleach plant filtrate.

30 10. A method as claimed in any of claims 3 to 9, wherein manganese is reduced by at least 70% of the original level in the bleach plant filtrate.

11. A method as claimed in any of claims 3 to 10, wherein the silica is reduced by at least 50% of the original level in the bleach plant filtrate.

35 12. A method as claimed in any of claims 3 to 11, wherein the silica is reduced by at least 70% of the original level in the bleach plant filtrate.

13. A method as claimed in any of claims 1 to 12, wherein the centrifugal cleaner is at least one forward cleaner.

40 14. A method as claimed in any of claims 1 to 13, where the centrifugal cleaner is a reverse cleaner.

15. The accept stream of any of claims 1 to 14, wherein said stream is circulated to the first bleach plant stage to wash the pulp.

45 16. The accept stream of any of claims 1 to 14, wherein said stream is circulated to any previous bleach plant stage to wash the pulp.

50 17. The accept stream of any of claims 1 to 14, wherein said stream is circulated to the last bleach plant stage to wash the pulp.