

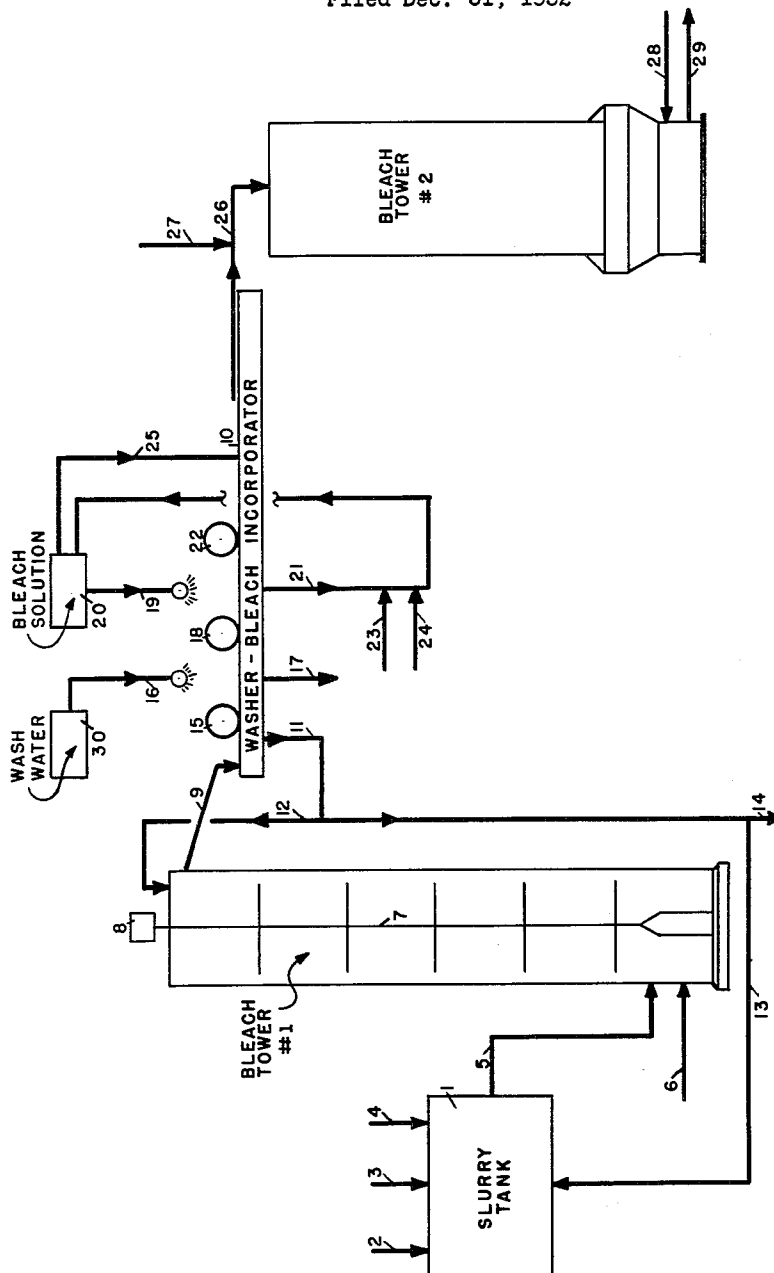
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CONTINUOUS BLEACHING PROCESS

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CONTINUOUS BLEACHING PROCESS

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This invention relates to the bleaching or refining of cellulosic material. In a specific aspect this invention relates to a continuous process for the bleaching or refining of cellulosic fibers.

Cellulosic materials, such as cotton linters or wood pulp, can be used in paper pulps and for the production of cellulose derivatives, such as cellulose ethers and esters, and prior to such uses the cellulosic materials are purified, bleached or refined to increase the α -cellulose content and to decrease the content of pentosans, lignin, coloring materials and other impurities. Various methods have been employed to purify, bleach or refine cellulosic materials. In these methods it is common practice to employ one or more sequences of steps in which the cellulosic material is treated first at low consistency and then at high consistency. For example, the conventional first stage of bleaching involves treatment of the unbleached cellulosic material in a low consistency slurry and in a second stage the chlorinated cellulosic material is treated with alkali and/or hypochlorite in a high consistency slurry. Modern practice is to use a continuous upflow tower for the low consistency treatment, one or more stages of continuous washing to remove the liquor from the low consistency treatment, a separate mixer for the incorporation of the exact amount of chemicals required for the second stage treatment, and a downflow high consistency tower for accomplishing the second stage of treatment. In these conventional processes, difficulty has been observed in obtaining uniform distribution of the small amount of chemicals for the second stage throughout the moist cellulosic material coming from the intermediate vacuum washers. Also, the separate washing and chemicals incorporation steps are time consuming and necessitate the use of separate equipment.

It is an object of this invention to provide a novel and improved procedure for purifying, bleaching or refining cellulosic materials.

It is another object of this invention to provide an improved continuous process for the purification, bleaching or refining of cellulosic materials involving a low consistency treatment, such as chlorination, followed by a high consistency treatment, such as alkaline extraction or hypochlorite bleaching.

Further and additional objects of this invention will be apparent from the detailed disclosure and the accompanying drawing.

It has been found that cellulosic materials, such as cotton linters, wood pulp, and the like, can be bleached or purified by a novel continuous process which involves the mixing of chlorine gas with an aqueous slurry of cellulosic material in an agitated contacting zone, followed by passage of chlorinated slurry upwardly through a reaction zone. The resulting slurry is then continuously passed to an incorporation zone where chlorination liquor is removed from the chlorinated cellulosic material, the resulting cellulosic material is washed with water, and the washed material is prepared for the sec-

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ond high consistency step by incorporation of an excess of an aqueous solution of a member of the group consisting of bleaching agents and alkaline extraction agents. The excess solution is continuously removed, and the resultant high consistency slurry is continuously passed to a second reaction zone where the cellulosic material is additionally purified by the prolonged action of the bleaching or extraction agent.

The process of this invention can be understood by reference to the accompanying drawing which is a diagrammatic procedure of a preferred method of carrying out this invention. In the description of this drawing and of the detailed process below a bleaching procedure involving a low consistency chlorination step and a high consistency hypochlorite bleach step will be described. However, it will be understood that, if desired, the hypochlorite bleach step can be replaced by a high consistency extraction step. If an extraction is employed, an extracting agent, instead of a hypochlorite bleaching agent, is incorporated in the cellulosic material prior to its introduction to the high consistency reaction zone.

Referring now to the accompanying drawing, cellulosic material, for example, wood pulp or digested cotton linters, enters slurry tank 1 via line 2 where the cellulosic material is slurried to a concentration within the range of 2% to 7% by weight with water entering via line 3. The exact slurry concentration for optimum operation will be determined by the fiber structure and fiber length of the cellulosic material. The preferred concentration is about 4.5% by weight. The aqueous slurry is pumped via line 5 to bleach tower #1. Chlorine gas is injected into line 5 or into the bottom of tower #1 via line 6 and is uniformly mixed with the aqueous slurry by agitation. If the amount of chlorine being added is small, it is customary to adjust the pH of the aqueous slurry to lower than 3.5 with a mineral acid, such as sulfuric acid, entering the slurry tank 1 via line 4. Although the chlorination step effected in tower #1 can be carried out without the use of means for agitating the reaction mixture, it is preferred to agitate the reaction mixture as it passes upwardly through the low consistency bleach tower. For that reason, bleach tower #1 is provided with agitator 7 operated by driving mechanism 8 to provide agitation along the line of upward flow of the slurried cellulosic material. In bleach tower #1 the chlorination is carried out for a period of 30 to 120 minutes at cold water temperature, and in the upper portion of the bleach tower the stock is diluted with recycled water to be described below.

A slurry containing from 1% to 2% by weight of cellulosic material is withdrawn from the upper portion of bleach tower #1 and passed via line 9 to a washing-bleach incorporation zone 10. Zone 10 is one of the important features of this invention since in this zone slurring liquid from bleach tower #1 is removed from the cellulosic material, and the cellulosic material is prepared for a second bleaching step by incorporating an excess of the second bleaching liquor followed by removal of the excess. The equipment for carrying out the essential operation in zone 10 can be a horizontal revolving circular foraminous plate or screen provided with means for spraying wash water and bleach solution onto the cellulosic material and optionally provided with press or squeeze rolls for assisting in the removal of liquid from the cellulosic material. It is also desirable to provide the lower portion of zone 10 with suitable suction means to assist in the removal of the liquid from the cellulosic material by withdrawal through the foraminous plate or screen. Slurried cellulosic material enters zone 10 via line 9 and chlorinating water is withdrawn via line 11. A portion of this chlorinating water is recycled

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to bleach tower #1 via line 12. A second portion of the chlorinating water is returned to slurry tank 1 via line 13 and any remaining chlorinating water is removed from the system via line 14. If desired, the separation of chlorinating water from cellulosic material can be assisted in zone 10 by the use of press roll 15. After removal of chlorinating water, fresh wash water from storage 30 is sprayed onto the cellulosic material via line 16 in order to displace chlorinating water remaining on the cellulosic material. Wash water and the displaced chlorinating water are then removed from the cellulosic material and withdrawn from the system via line 17. Optionally, press roll 18 is employed to assist in the separation of wash water and remaining chlorinating water from the cellulosic material. An excess of sodium hypochlorite bleach liquor is then sprayed onto the washed cellulosic material. The bleach liquor enters zone 10 via line 19 from storage 20. The liquor from line 19 displaces wash water from the cellulosic material and the excess of this liquor diluted by displaced wash water is withdrawn from the cellulosic material via line 21. Press roll 22 can be used, if desired, to assist in the separation of the bleach solution and displaced wash water from the cellulosic material. It is an important feature of this invention that hypochlorite bleach solution in an amount in excess of that required for subsequent treatment is added to the cellulosic material via line 19. For example, from 10 to 30 parts of bleach solution per part of cellulosic material can be added via line 19. Any excess hypochlorite bleach solution is removed via line 21. The application of the excess bleach solution insures saturation of the cellulosic material with bleach solution so that the liquor is incorporated thoroughly and uniformly throughout the cellulosic material. Fresh chlorine and aqueous sodium hydroxide are introduced to line 21 via lines 23 and 24, respectively, in order to refortify the dilute bleach solution and the resulting refortified solution is returned to storage 20. The accumulation of bleach solution in storage unit 20 is prevented by withdrawing as overflow an amount substantially equivalent in weight to the added chlorine and aqueous sodium hydroxide solution via line 25, and the overflow bleach solution is thus added to the cellulosic material containing hypochlorite bleach solution prior to its removal from zone 10.

It is evident from the above discussion that the following operations are carried out in zone 10: (a) the removal of chlorinating water from the cellulosic material; (b) the application of a fresh water wash to the cellulosic material; and (c) the addition of a hypochlorite bleach solution to the washed cellulosic material. In addition to these operations the diluted hypochlorite bleach solution is refortified with chlorine gas and aqueous sodium hydroxide to restore it to its original concentration prior to its recycle to zone 10.

Cellulosic material wetted with hypochlorite bleach solution is continuously removed from zone 10 via line 26 and thus passed to tower #2. It is preferred to employ a pin mixer or similar apparatus in line 26 in order to restore the cellulosic material to its fibrous form. Steam can be sparged onto the cellulosic material via line 27 to heat the material prior to its entrance into tower #2. The material containing from 4 to 8 parts by weight of liquor per part of material enters the top of tower #2 where it reacts for a period of 20 to 120 minutes. The temperature will ordinarily be between 120° and 200° F. for the usual hypochlorite bleach or alkaline extraction. It will be understood that the conditions prevailing in tower #2 can be varied within rather wide limits in order to obtain the desired purification of the cellulosic material. During the bleaching operation the cellulosic material passes downwardly in a mass through tower #2 and the reacted material is subsequently removed from the bottom of tower #2 by dilution or mechanical means. The reacted material is then removed via line 29 and it can

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then be subjected to any necessary washing and drying steps prior to its use in paper, in the production of cellulose derivatives, and the like. An effective tower for use in the second high consistency step of this process is described in U. S. Patent 2,730,888. This type of tower is quite effective for obtaining uniform treatment of the cellulosic material and uniform and efficient removal of the reacted material from the tower.

When bleaching is the objective of the high consistency stage of the process, sodium hypochlorite is the preferred bleaching agent for use in bleach tower #2. However, it will be understood that other bleaching agents, such as the alkaline earth metal hypochlorites, the alkali peroxides, and alkali metal hypochlorites other than sodium hypochlorite can be used. In most instances the aqueous hypochlorite bleach will be added in an amount equivalent to from 0.5% to 2.0% active chlorine based on the cellulosic material. When the high consistency stage of this process is used for alkaline extraction, the conditions will vary depending on whether the objective of this step is the removal of chlorinated colored materials or whether it is desired to raise the α -cellulose content by removal of noncellulosic carbohydrate materials. For the removal of colored materials as an aid in bleaching, caustic alkali equivalent to 2% to 8% based on the cellulosic material is used at temperatures of 120° to 160° F. Such conditions are employed in the bleaching of kraft wood pulps, cotton linters, and low α -sulfite pulps. For the production of higher α -sulfite pulps, caustic in amounts equal to as high as 15%, based on the pulps, and temperatures as high as 200° F. can be used. Any of the commonly used extraction agents can be employed in tower #2 with sodium hydroxide being the preferred reagent. Further modifications and alternative procedures within the scope of this invention will be apparent to those skilled in the art from the above disclosure.

What I desire to claim and protect by Letters Patent is:

1. The method for the continuous purifying of cellulose fibers which comprises, in combination, forming a dilute aqueous slurry of said fibers, continuously passing aqueous slurry and chlorine gas to a lower portion of a contacting zone and therein contacting said fibers and chlorine in a first bleaching step, continuously withdrawing a slurry of fibers and bleach liquor from an upper portion of said contacting zone, continuously passing withdrawn slurry to an incorporation zone, removing bleach liquor from said fibers in said incorporation zone and recycling removed bleach liquor to said contacting zone, in said incorporation zone washing remaining bleach liquor from said fibers with water and adding an aqueous solution of a member of the group consisting of bleaching agents and alkaline extraction agents to the washed fibers in an amount in excess of the amount required for the second reaction step, in said incorporation zone removing excess aqueous solution diluted with wash water from said fibers, refortifying diluted excess aqueous solution, recycling refortified aqueous solution to said incorporation zone for incorporation in additional cellulose fibers, throughout their passage through said incorporation zone said fibers being in the form of a continuous sheet or cake, continuously withdrawing cellulose fibers wetted with the amount of aqueous solution required for the second reaction step from said incorporation zone, and continuously reacting withdrawn cellulose fibers with wetting aqueous solution in a second reaction step.

2. The method for the continuous bleaching of cotton linters which comprises, in combination, forming a dilute aqueous slurry of said linters, continuously passing aqueous slurry and chlorine gas to a lower portion of an agitated contacting zone and therein contacting said linters and chlorine in a first bleaching step, continuously withdrawing a slurry of linters and bleach liquor from an upper portion of said contacting zone, continuously

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passing withdrawn slurry to a bleach incorporation zone, removing bleach liquor from said linters in said bleach incorporation zone and recycling removed bleach liquor to an upper portion of said contacting zone, in said bleach incorporation zone washing remaining bleach liquor from said linters with water and adding aqueous sodium hypochlorite bleach solution to the washed linters in an amount in excess of the amount required for the second bleaching step, in said bleach incorporation zone removing excess hypochlorite bleach solution diluted with wash water from said linters, refortifying said diluted bleach solution with additional sodium hypochlorite, recycling refortified bleach solution to said bleach incorporation zone for incorporation in additional cotton linters, throughout their passage through said incorporation zone said fibers being in the form of a continuous sheet or cake, continuously withdrawing cotton linters wetted with the amount of sodium hypochlorite bleach solution required for the second bleaching step from said bleach incorporation zone, and continuously bleaching withdrawn cotton linters with wetting sodium hypochlorite bleach solution in a second bleaching step.

3. The method for the continuous bleaching of cotton linters which comprises, in combination, forming a dilute aqueous slurry of said linters, continuously passing aqueous slurry and chlorine gas to a lower portion of an agitated contacting zone and therein contacting said linters and chlorine in a first bleaching step, continuously withdrawing a slurry of linters and bleach liquor from an upper portion of said contacting zone, continuously passing withdrawn slurry to an incorporation zone, removing bleach liquor from said linters in said incorporation zone and recycling removed bleach liquor to an upper portion of said contacting zone, in said incorporation zone washing remaining bleach liquor from said linters with water and adding aqueous solution of an alkaline extraction agent to the washed linters in an amount in excess of the amount required for the subsequent extraction step, in said incorporation zone removing excess extraction agent solution diluted with wash water from said linters, refortifying said diluted solution with additional extraction agent, recycling refortified extraction solution to said incorporation zone for incorporation in additional cotton linters, throughout their passage through said incorporation zone said fibers being in the form of a continuous sheet or cake, continuously withdrawing cotton linters wetted with the amount of extraction solution required for the subsequent extraction step from said incorporation zone, and continuously reacting withdrawn cotton linters with wetting extraction solution in an extraction step.

4. The method for the continuous bleaching of cotton linters which comprises, in combination, forming a dilute aqueous slurry of said linters having a concentration of linters within the range of 2% to 7% by weight, continuously passing aqueous slurry and chlorine gas to a lower portion of an agitated contacting zone and therein contacting said linters and chlorine for a period of 30 to 120 minutes in a first bleaching step, continuously withdrawing a slurry of linters and bleach liquor from an upper portion of said contacting zone, continuously passing withdrawn slurry to a bleach incorporation zone, removing bleach liquor from said linters in said bleach incorporation zone and recycling removed bleach liquor to an upper portion of said contacting zone, in said bleach incorporation zone washing remaining bleach liquor from said linters with water and adding aqueous sodium hypochlorite bleach solution to the washed linters in an amount in excess of the amount required for the second bleaching step and within the range of 10 to 30 parts per part of linters, in said bleach incorporation zone removing excess hypochlorite bleach solution diluted with wash water from said linters, refortifying said diluted bleach solution with additional sodium hypochlorite, recycling refortified bleach solution to said bleach incorporation

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zone for incorporation in additional cotton linters, throughout their passage through said incorporation zone said fibers being in the form of a continuous sheet or cake, continuously withdrawing cotton linters wetted with an amount of sodium hypochlorite bleach solution within the range of 4 to 8 parts by weight of solution per part of linters from said bleach incorporation zone, and continuously bleaching withdrawn cotton linters for a period of 20 to 120 minutes at a temperature of 120°–200° F. with wetting sodium hypochlorite bleach solution in a second bleaching step.

5. The method for the continuous bleaching of cotton linters which comprises, in combination, forming a dilute aqueous slurry of said linters having a concentration of linters within the range of 2% to 7% by weight, continuously passing aqueous slurry and chlorine gas to a lower portion of an agitated contacting zone and therein contacting said linters and chlorine for a period of 30 to 120 minutes in a first bleaching step, continuously withdrawing a slurry of linters and bleach liquor from an upper portion of said contacting zone, continuously passing withdrawn slurry to an incorporation zone, removing bleach liquor from said linters in said incorporation zone and recycling removed bleach liquor to an upper portion of said contacting zone, in said incorporation zone washing remaining bleach liquor from said linters with water and adding aqueous sodium hydroxide to the washed linters in an amount in excess of the amount required for the subsequent extraction step and within the range of 10 to 30 parts per part of linters, in said incorporation zone removing excess aqueous sodium hydroxide diluted with wash water from said linters, refortifying said diluted aqueous sodium hydroxide with additional sodium hydroxide, recycling refortified sodium hydroxide solution to said incorporation zone for incorporation in additional cotton linters, throughout their passage through said incorporation zone said fibers being in the form of a continuous sheet or cake, continuously withdrawing cotton linters wetted with an amount of aqueous sodium hydroxide within the range of 4 to 8 parts by weight of aqueous sodium hydroxide per part of linters from said incorporation zone, and continuously reacting withdrawn cotton linters with wetting sodium hydroxide solution for a period of 20 to 120 minutes at a temperature of 120°–200° F. in an extraction step.

6. The method for separating cellulose fibers from an aqueous chlorine-containing bleach liquor and for incorporating aqueous solution of a member of the group consisting of bleaching agents and alkaline extraction agents in the separated cellulose fibers which comprises, in combination, passing a slurry of cellulose fibers in chlorine-containing bleach liquor to an incorporation zone, removing bleach liquor from said fibers in said incorporation zone, washing remaining bleach liquor from said fibers with water in said incorporation zone, adding aqueous solution of a member of the group consisting of bleaching agents and alkaline extraction agents to washed fibers in said incorporation zone in excess of the amount required for subsequent reaction of said fibers to displace wash water remaining on said fibers, removing excess aqueous solution diluted with displaced wash water from said fibers in said incorporation zone, refortifying said diluted excess aqueous solution with additional solution, recycling refortified solution to said incorporation zone for incorporation in additional cellulose fibers, throughout their passage through said incorporation zones said fibers being in the form of a continuous sheet or cake, and withdrawing from said bleach incorporation zone cellulose fibers wetted with aqueous solution.

7. The method for separating cotton linters from an aqueous chlorine-containing bleach liquor and for incorporating aqueous sodium hypochlorite bleach solution in the separated cotton linters which comprises, in combination, passing a slurry of cotton linters in said bleach liquor

to a bleach incorporation zone, removing bleach liquor from said linters and washing remaining bleach liquor from said linters in said bleach incorporation zone, adding aqueous sodium hypochlorite bleach solution to washed linters in said bleach incorporation zone in excess of the amount required for subsequent bleaching of said linters to displace wash water remaining on said linters, removing excess hypochlorite bleach solution diluted with displaced wash water from said linters in said bleach incorporation zone, refortifying said diluted excess hypochlorite bleach solution with additional sodium hypochlorite bleach, recycling refortified bleach solution to said bleach incorporation zone for incorporation in additional cotton linters, throughout their passage through said incorporation zone said fibers being in the form of a continuous sheet or cake, and withdrawing from said

bleach incorporation zone cotton linters wetted with sodium hypochlorite bleach solution.

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