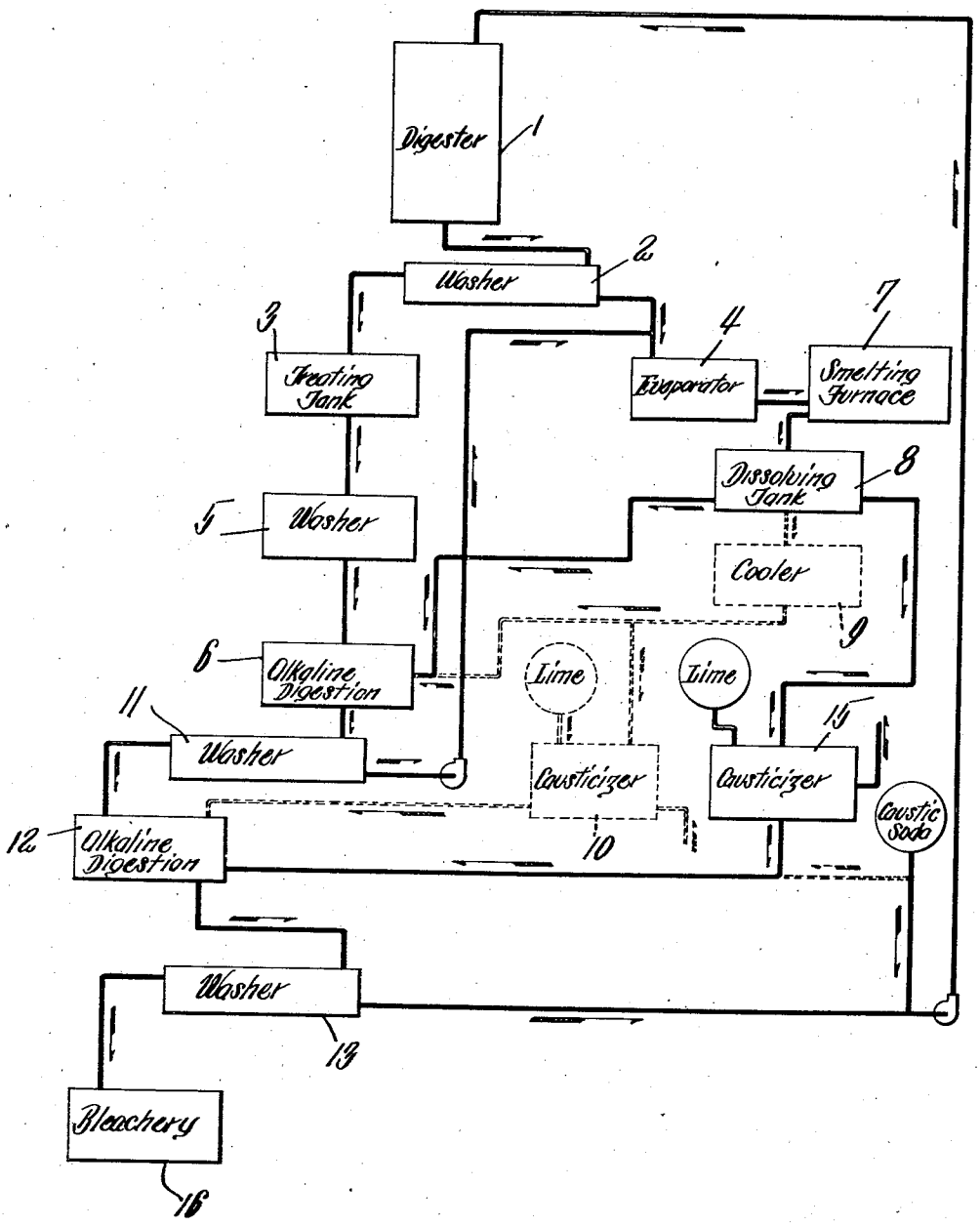
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PROCESS OF REFINING RAW CELLULOSE PULP

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PROCESS OF REFINING RAW CELLULOSE PULP

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This invention relates to a process of refining raw or unbleached cellulose pulps such as kraft, soda, or sulphite, its object being the production of a white product suitable for use as a high grade raw material for papermaking and for the preparation of cellulose derivatives.

Raw cellulose pulps resulting from the various processes of fiber liberation vary considerably, their physical characteristics and compositions depending upon the fiber-liberating process by which they were produced and the conditions under which fiber liberation was effected. When such pulps are produced from raw cellulosic material such as wood, they contain a substantial amount of ligneous and other coloring matter and less resistant celluloses, the usual kraft pulp being quite high in lignin and accordingly requiring such a large amount of bleach to be whitened as to undergo physical and chemical degradation during the bleaching operation. It has been found that if raw pulp such as kraft is treated with lignin-reactive reagents, such as chlorine water or hypochlorite or bisulphite liquors, to effect a thorough reaction on the ligneous groups in the pulp, then washed free of reagent and soluble reaction products, and then digested in an alkaline liquor of suitable strength and under suitable temperature conditions, the resulting product is readily bleachable to whiteness without injury to the pulp. The lignin-reactive reagents mentioned evidently react on the ligneous groups present in the pulp to produce water-soluble products, which are removed during washing, and water-insoluble products, which are soluble in alkaline liquors.

The alkaline liquor, depending on its alkalinity and temperature, may serve not only to remove water-insoluble acidic products, but also less resistant celluloses and other non-alpha cellulose impurities present in the pulp.

I have found that raw pulp, particularly after treatment with the lignin-reactive reagents described, contains non-alpha cellulose components which are readily soluble in liquors of comparatively low alkalinity, and non-alpha cellulose components which are dissolved in liquors of comparatively high alkalinity only. In accordance with the process of the present invention, therefore, raw pulp which has been treated with such lignin-reactive reagents is first digested in an alkaline liquor of comparatively low alkalinity, and then in a liquor of comparatively high alkalinity. The initial alkaline liquor may be a sodium carbonate solution or a milk of lime suspension, as I have found that such liquors are capable of readily removing water-insoluble reaction products formed during the previous treatment with lignin-reactive reagent. Heretofore, it has been proposed to digest pulp which has previously been treated with a lignin-reactive reagent such as chlorine water, in alkaline liquors of comparatively high alkalinity,—for instance, solutions of caustic soda and/or sodium sulphide, with the result that reaction products present in the pulp after washing consumed a substantial amount of such alkali. Such reaction products may, however, be removed with sodium carbonate and/or a milk of lime liquor, whereupon the more difficultly soluble non-alpha cellulose components in the pulp may be removed by digesting the pulp in solutions of stronger alkalies, such as caustic soda and/or sodium sulphide. In a kraft mill, the process of the present invention makes possible a saving of lime, as sodium carbonate is produced in large quantity during the smelting of the inorganic content of the spent cooking liquor. The sodium carbonate occurs in the smelt in admixture with sodium sulphide, but it may be readily removed from the sodium sulphide as by fractional crystallization, and used in the preparation of an alkaline liquor for initially digesting the pulp, thereby effecting a saving of the lime customarily used in causticizing such sodium carbonate. The mother liquor, which is rich in sodium sulphide, after causticization of residual sodium carbonate therein, if desired, may be used in preparing the liquor for the second alkaline digestion of the pulp. When a concentrated liquor at comparatively low

temperatures is used for the second alkaline digestion to produce a final product of high alpha cellulose content, the liquor obtained from such digestion has a high content of unspent alkali, and accordingly may be used to advantage, after fortification with fresh alkali, if desired, for the preparation of a cooking liquor for liberating fiber from raw wood.

The accompanying drawing represents a flow sheet in which the steps of the process of the present invention are interrelated with a process of producing kraft pulp.

Referring to the drawing, 1 represents a digester in which the cooking of raw cellulosic material such as chipped wood may be effected in a kraft cooking liquor containing, say, 2 to 2.4 pounds of active alkali, calculated as Na_2O per cubic foot, and 40% or higher sulphidity. Cooking may be carried out for a four-hour period at a maximum temperature of 335° F. The liberated pulp may be delivered into a washer 2, the washed pulp being passed into a treating tank 3, and the cooking liquor being passed into an evaporator 4. The pulp may be treated with one or more lignin-reactive reagents. For instance, the pulp may be treated at a consistency of from 2% to 8% in chlorine water containing, say, 3% to 8% chlorine, based on dry pulp, the time of contact ranging from 20 to 200 minutes, and the temperature from 50° to 90° F., depending upon the characteristics of the raw pulp and the color and other characteristics desired in the final product. The higher the chlorine usage, the greater the advantages accruing from the present process, as more reaction product is removed in the weak alkaline solution. If desired, a bisulphite liquor may be used in lieu of the chlorine water, the bisulphite reacting to form lignone-sulphonic reaction products, part of which are soluble in water and part of which are soluble in the weak alkaline solution. The pulp may be treated at a consistency of from 2% to 8% in a boiling bisulphite liquor, preferably a sodium bisulphite liquor containing about 1% free and 1% combined SO_2. It may be desirable to combine a chlorine water treatment and a bisulphite liquor treatment, in which case it is preferable to chlorinate the stock first, as some of the chlorinated products are soluble in hot bisulphite liquor. After treatment with a lignin-reactive reagent, the pulp may be delivered into a washer 5, wherein the reagent together with soluble reaction products is washed from the pulp, whereupon it may be delivered into a tank 6 for digestion in weak alkaline liquor. A ½% to 1% sodium carbonate solution at boiling temperature may be used, digestion being continued for about two to four hours, depending upon the strength of the solution. The sodium carbonate may be derived from the smelt of the inorganic compounds of the spent kraft cooking liquor. As shown on the drawing, after the spent kraft liquor has been concentrated in the evaporator 4, it may be delivered into a furnace 7, wherein its organic content may be burned and its inorganic content smelted in a reducing atmosphere to produce a sodium carbonate-sodium sulphide smelt. The hot smelt may be delivered into a body of water in a tank 6, to produce a hot smelt solution, which may be diluted to the desired strength and passed into the tank 6 for the digestion of the pulp, in which case the alkaline liquor consists of a sodium carbonate-sodium sulphide solution. Preferably, however, as indicated in dotted outline, the hot concentrated smelt solution is passed through a cooler 9 designed to cause crystallization of sodium carbonate only to take place, the crystallized sodium carbonate being delivered into the tank 6 to form a solution of the strength desired for the digestion of the pulp. The mother liquor, rich in sodium sulphide, may be passed into a tank 10, from which it may be withdrawn, preferably after causticization of its residual sodium carbonate content with lime, for the second alkaline digestion of the pulp. The second alkaline digestion is performed after the pulp associated with the spent liquor from the first digestion has been passed through a washer 11 for the removal of spent cooking liquor and the soluble reaction products. The liquor from the washer 11 may be commingled with the liquor from the washer 2, the mixture of spent liquors being evaporated and passed into the furnace 7, as previously described. The concentration of alkali and temperature during the second digestion depends upon the characteristics desired in the final product. For instance, if it is desired to produce a product of high alpha cellulose content, such digestion may be carried out in a liquor containing 5% to 6% alkali calculated as NaOH in the form of NaOH and/or Na_2S, at a temperature of about 20° C. Such a liquor effects a substantial removal of pentosans and less-resistant celluloses from the pulp, the alpha cellulose content of the pulp being increased correspondingly. The pulp resulting from such digestion is further characterized by a high strength, tear resistance, folding endurance, and softness. It has a pentosan content ranging from 2% to 5%, as compared with a pentosan content of 8% to 10% in the original kraft pulp. If a product intended primarily for papermaking is desired, as much diluter alkaline liquor may be used at relatively high temperatures, say, 212° F., in which case less pentosans will be removed. When a concentrated liquor is used, the spent cooking liquor has a high

content of unspent or active alkali. Accordingly, after the pulp has been passed through a washer 13 preparatory to final bleaching, the liquor from the washer may be used as the cooking liquor in the digester 1, fresh caustic soda being added to the liquor before such use, if desired. If, however, a comparatively dilute liquor has been employed for such second alkaline digestion, and the liquor contains little active alkali after digestion, it may be mixed with the liquors from the washers 11 and 2 into the evaporator 4, whereupon the mixed liquors may be passed into the smelting furnace.

As previously stated, the initial alkaline digestion of the pulp may be effected with a lime liquor in lieu of a sodium carbonate solution. Such a procedure also makes possible a saving, as lime is a much less expensive chemical than are sodium base compounds such as sodium carbonate, caustic soda, or sodium sulphide. The pulp may in such case be digested as a 6% suspension in an 8% to 10% milk of lime liquor at 212° F. for four hours. After digestion has been completed, the major portion of lime will have gone into solution, but there will be residual lime in suspension. The liquor may be removed from the pulp by washing, it being preferable to use wash water initially rendered slightly acidic as with acetic acid, to ensure the removal of the lime. When digestion is effected in a lime liquor, the smelt solution may be delivered into a tank 15, wherein its sodium carbonate content may be causticized with lime. The causticized liquor may be used for the second digestion of the pulp, additional caustic soda being added thereto, if desired. After the second alkaline digestion of the pulp and washing, it is readily bleachable without suffering injury, as indicated on the drawing, the pulp being delivered into a tank 16, wherein it may be bleached as ordinarily with calcium or sodium hypochlorite bleach liquor. Preferably, bleaching is effected in a distinctly alkaline bleach liquor, this operation being carried out in several steps with washings between the steps, as this ensures no injury being done to the pulp.

A process such as hereinbefore described may also be advantageously practised in connection with the production of soda pulp. The smelt liquor in a soda mill consists of a sodium carbonate solution and is ordinarily causticized. In accordance with the process of the present invention, a portion of the sodium carbonate smelt solution may be used in the initial alkaline digestion of the pulp, thereby effecting a saving of the lime ordinarily used in causticization. The rest of the smelt solution may be causticized as usual and employed for the second alkaline digestion.

The process of the present invention having thus been described along with various applications thereof, it should be evident that various changes and modifications might be made therein without departing from the spirit or scope of invention as defined by the appended claims.

I claim:

1. A process which comprises treating raw cellulose pulp with a lignin-reactive reagent, washing the pulp, digesting the pulp in a weakly alkaline liquor to dissolve certain ligneous reaction products therefrom, and then digesting the pulp in a strongly alkaline liquor to dissolve other non-alpha cellulose components therefrom.

2. A process which comprises treating raw cellulose pulp with a lignin-reactive reagent, digesting the pulp in a sodium carbonate solution to dissolve certain ligneous reaction products therefrom, and then digesting the pulp in a strongly alkaline liquor to remove other non-alpha cellulose components therefrom.

3. A process which comprises chlorinating raw cellulose pulp, digesting the chlorinated pulp in a sodium carbonate solution to dissolve certain chlorine-lignin reaction products therefrom, and then digesting the pulp in a caustic soda solution to dissolve other non-alpha cellulose components therefrom.

4. A process which comprises treating raw kraft pulp with chlorine water, digesting the chlorinated pulp at elevated temperature in a sodium carbonate solution to dissolve certain chlorine-lignin reaction products therefrom, and then digesting the pulp at about room temperature in a strong solution of caustic soda and sodium sulphide to dissolve other non-alpha cellulose components therefrom.

5. A process which comprises cooking raw cellulosic material in a cooking liquor containing sodium-sulphur compounds to effect fiber liberation, separating the spent liquor from the resulting pulp, smelting the inorganic constituent of the spent liquor to produce sodium carbonate and sodium sulphide, separating sodium carbonate from the rest of the smelt, digesting the pulp in a solution of the separated sodium carbonate, and then digesting the pulp in a solution of the rest of the smelt.

6. A process which comprises cooking raw cellulosic material in a cooking liquor containing sodium-sulphur compounds to effect fiber liberation, separating the spent liquor from the resulting pulp, smelting the inorganic constituent of the spent liquor to produce sodium carbonate and sodium sulphide, forming an aqueous smelt solution, crystalizing sodium carbonate from the smelt solution, digesting the pulp in a solution of the separated sodium carbonate, causticizing the rest of the smelt solution,

and digesting the pulp in a solution of the causticized smelt solution.

7. A process which comprises cooking raw cellulosic material in a kraft liquor to effect fiber liberation, separating the spent liquor from the resulting pulp, smelting the inorganic content of the spent liquor to produce a smelt containing sodium carbonate and sodium sulphide, selectively crystallizing sodium carbonate from the smelt solution, treating the raw pulp with a lignin-reactive reagent, digesting the pulp in a solution of the crystallized sodium carbonate, and then digesting the pulp in the smelt mother solution.

8. A process which comprises digesting preliberated cellulose pulp in a milk of lime liquor to dissolve non-alpha cellulose components soluble in such a liquor, and then digesting the pulp in a much stronger alkaline liquor to dissolve other non-alpha cellulose components therefrom.

9. A process which comprises digesting preliberated cellulose pulp in a milk of lime liquor to dissolve non-alpha cellulose components soluble in such a liquor, washing the pulp with water initially in an acid condition to ensure the removal of lime, and then digesting the pulp in a solution of caustic soda to dissolve other non-alpha cellulose components therefrom.

10. A process which comprises digesting preliberated cellulose pulp in a sodium carbonate solution to dissolve non-alpha cellulose components soluble in such a solution, and then digesting the pulp in a much stronger alkaline liquor to dissolve other non-alpha cellulose components therefrom.

11. A process which comprises digesting preliberated cellulose pulp in a sodium carbonate solution to dissolve non-alpha cellulose components soluble in such liquor, and then digesting the pulp in a caustic soda solution to dissolve other non-alpha cellulose components therefrom.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.