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PROCESS OF REFINING ALKALINE PULPS

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This invention has relation to the production of a white high alpha cellulose fiber, i. e., a fiber containing a high percentage of alpha or resistant cellulose, possessing high strength and tear qualities, from a pulp resulting from the digestion of wood chips in an alkaline liquor, and more particularly in a kraft or sulphate liquor.

Pulp produced by an alkaline process of digestion, such as kraft, is especially characterized by its brownish color, doubtless arising from the considerable amount of ligneous and other coloring substance and less-resistant celluloses contained therein, and also by its quality of high strength, which renders such pulp very valuable commercially for use in the production of strong papers, viz., wrapping and bag papers, where the characteristic of color is relatively unimportant. Such pulp, however, is almost exclusively restricted to the manufacture of wrapping or bag papers, and ordinarily cannot be employed for purposes where a pulp of high purity or high alpha cellulose content is required; nor is it ordinarily used in the manufacture of writing papers where a white pulp is desired.

Moreover, such pulp is exceedingly difficult to bleach. While it is possible to bleach such pulp with an excessive amount of bleach, say, about 50% of 35% bleach ($CaOCl_2$) based on the weight of dry pulp, the strength of the resultant product is low and its alpha cellulose content thereby suffers a material decrease by conversion or degradation into oxycelluloses so that the commercial value of the product is lowered. This difficulty in bleaching may be directly traced to the high percentage of ligneous and other coloring substance remaining in the kraft pulp after the alkaline digestion.

In the course of research work, I have discovered, generally stated, that if an alkaline cooked pulp, such as kraft, is treated with an oxidizing or bleaching agent in the presence of a large excess of a strongly alkaline liquor such as a solution of caustic soda, the pulp is purified or freed from less-resistant celluloses, ligneous and other coloring impurities, without perceptible injury to the alpha cellulose content thereof, and is thereby converted into a refined and easily bleachable pulp which is high in alpha or resistant-cellulose and, because of its characteristics, is adapted for a great variety of uses which will subsequently be pointed out.

The kraft pulp employed in the present invention may be produced as ordinarily by the digestion of wood chips in a digester under the requisite time, temperature, and pressure conditions in accordance with modern practice, in the usual kraft or sulphate liquor, containing sodium sulphide, sodium hydroxide, and but an adventitious or relatively slight amount of sodium sulphate. Spruce, hemlock, pine, straw, or any other raw cellulosic material suitable for the production of kraft pulp may be utilized. After the necessary period of digestion, the digester contents are blown and the kraft pulp is washed and separated from its accompanying black spent digesting liquor. The washed pulp is then preferably screened to remove shives, specks and other contaminating matter and is then thickened to the consistency desired for admixture with the alkaline liquor containing the oxidizing agent. The thickened pulp is then intimately mixed with a concentrated solution of caustic soda containing a relatively small proportion of bleach or oxidizing agent, and is treated therewith, say at room temperature (30° C.) for a sufficient period of time to permit the bleach and alkali to react with and remove the less-resistant celluloses, ligneous and other coloring impurities contained in the pulp. In carrying out the treatment or purification, too high a concentration of caustic soda must be avoided, since otherwise the fiber will become mercerized, such mercerization tending to lower the strength of the pulp, and in such case subsequent beating merely macerating or shredding the fiber without effecting its hydration so that paper made therefrom is extremely weak. On the other hand, if too low a concentration of caustic soda is employed, sufficient reaction of the impurities contained in the pulp with the caustic soda solution will not take place and the resulting pulp will not be sufficiently

high in alpha cellulose. In other words, the concentration of caustic soda employed in the alkaline treatment is preferably maintained between two limits, namely, between a maximum concentration at which mercerization takes place and a minimum concentration at which a sufficient purification of the pulp is not effected. These limits will vary somewhat, depending upon the temperature of treatment employed and differences in characteristics of the initial raw stock. After the purification, the pulp, containing about 93% alpha cellulose, is washed free from its treating liquor and may be bleached to a high degree of whiteness with a moderate amount of hypochlorite bleach without materially injuring its strength or decreasing its alpha cellulose content. If a pulp of maximum whiteness is desired, the bleached pulp may be superbleached in a chlorine solution.

While I have indicated that the process is ordinarily carried out at a relatively low temperature, e. g., room temperature, it may be stated that lower than room temperature (say, 0° C. to 25°C.) may be employed to effect the purification of the pulp. Likewise higher temperature, for example, 30° C. to 75° C., and a correspondingly suitable concentration of caustic soda, may be employed in such treatment. For optimum results in respect to alpha cellulose content in a finished product prepared from a representative kraft stock at a treating temperature of about 30° C., the minimum and maximum concentrations in a stock suspension of about 5% to 6% are, respectively, about 100% and 175% caustic soda based on the weight of dry fiber. The concentration of bleach may vary from 5% to 20% of 35% bleach (either NaOCl or $CaOCl_2$) based on the dry weight of pulp. If desired, where a soluble bleach such as sodium hypochlorite (NaOCl) is used, the bleach may be made by introducing chlorine (Cl_2) into the treating liquor until the desired concentration of bleach is formed therein.

While I am unable to explain precisely what occurs in the purification of the kraft pulp, it may be that the oxidation of the difficultly removable ligneous or coloring substance contained in the kraft pulp in the presence of a very large excess of caustic soda is sufficiently retarded to prevent the attack of the alpha cellulose content thereof while permitting sufficient action on the non-alpha cellulose constituents. Not only, however, does the large excess of strong caustic soda solution regulate the degree of the reaction of the oxidizing agent to permit only a selective reaction between it and the less-resistant or easily reactable substances, but also reacts with such substances and dissolves out from the pulp products of both reactions. In any event, whatever the action be underlying the purification of the pulp, it may be stated that equivalent results are not achieved by subjecting the kraft pulp to a preliminary oxidizing reaction and then treating the pulp with an alkaline solution; or by first treating the pulp with an alkaline solution and then subjecting the treated pulp to an oxidizing reaction. That is to say, the results obtained by practicing the process of the present invention are dependent upon the treatment of the kraft pulp with the bleaching or oxidizing agent while in the presence of a large excess of alkaline agent, to supplement as well as to regulate the oxidizing reaction.

The alkaline oxidizing treatment is carried out, preferably while stirring or agitating the pulp so as to promote a uniform reaction, say at room temperature, for about five to six hours, depending upon the characteristics of the particular stock undergoing treatment. The resultant pulp, if the treatment is carried out under the optimum conditions stated, will contain from 93% to 97% alpha cellulose. The pulp may be separated and washed substantially free from alkaline and entrained products of the reaction, by passage through a counter-current washer, and the spent liquor thus separated from the pulp will not only contain the various organic impurities removed from the pulp, but a very high percentage of residual unconsumed caustic soda. The larger portion of spent liquor is preferably re-employed in subsequent treatment of other kraft pulp, and the chemicals in the remainder of such spent liquor may be recovered.

The washed pulp is of light color, not much different in appearance from the color of ordinary unbleached sulphite pulp, and, as stated, contains 93% to 97% alpha cellulose.

Since the treatment has removed from the original kraft pulp a substantial amount of ligneous and other coloring substance, together with less-resistant celluloses, the removal of such bleach-consuming constituents allows the pulp to be bleached with materially less hypochlorite or bleach than is required before such treatment to produce a pure white product, and thus the bleaching operation may be effected without injury to the fiber strength of the pulp or reduction of its alpha cellulose content. Ordinarily a bleaching treatment at a stock consistency of about 10% to 16% with about 5% to 10% of 35% lime bleach ($CaOCl_2$), based on the dry weight of pulp, is sufficient to bring the pulp up to a whiteness equivalent to or approximating that of ordinary bleached sulphite pulp.

The stock thus bleached may be improved in color by a superbleaching operation, which may be carried out at a stock consistency of from 4% to 6%; with about .2% to .5% chlorine based on the weight of dry fiber.

Due to its high alpha cellulose content,

rived from an alkaline digestion of wood chips, which process comprises treating such pulp at low temperature with a liquor containing bleach and a preponderant amount of caustic soda, said caustic soda being present in amount sufficient to react with non-alpha cellulose constituents contained in such pulp but insufficient to effect a substantial mercerization of the alpha cellulose content thereof, washing the treated pulp, and bleaching the washed pulp at a stock consistency of about 10% to 16% with about 5% to 10% of 35% lime bleach based on the dry weight of pulp.

7. In the production of a white fiber characterized by its high alpha or resistant cellulose content, a process which comprises treating alkaline digested wood pulp, at low temperature, with a liquor containing bleach and a preponderant amount of caustic soda, said caustic soda being present in amount sufficient to react with non-alpha cellulose constituents contained in such pulp but insufficient to effect a substantial mercerization of the alpha cellulose content thereof, washing the treated pulp, bleaching the washed pulp at a stock consistency of about 10% to 16% with about 5% to 10% of 35% lime bleach based on the dry weight of pulp, and superbleaching said bleached pulp at a stock consistency of from 4% to 6% with about .2% to .5% chlorine based on the dry weight of pulp.

8. In the production of a white fiber characterized by its high alpha or resistant cellulose content, a process which comprises treating alkaline digested wood pulp at a stock consistency of about 5% to 6% and at about room temperature for about five or six hours, with a liquor containing 5% to 20% of 35% bleach ($CaOCl_2$) and about 100% to 175% caustic soda, based on the weight of dry pulp, washing the treated pulp, bleaching the washed pulp at a stock consistency of about 10% to 16% with about 5% to 10% of 35% lime bleach based on the dry weight of pulp, and superbleaching said bleached pulp at a stock consistency of from 4% to 6% with about .2% to .5% chlorine based on the dry weight of pulp.

9. In the production of fiber characterized by its high alpha or resistant cellulose content, a step which comprises treating alkaline digested wood pulp in a liquor containing an oxidizing agent and caustic soda, said liquor containing about 5% to 10% caustic soda.

10. A process which comprises treating wood pulp in a liquor containing both a preponderating proportion of caustic soda and a smaller but substantial proportion of an oxidant until the non-alpha cellulose content of such pulp has been largely dissolved, and bleaching the treated pulp.

11. In the production of fiber characterized by its high alpha cellulose content, that step which comprises treating an alkaline digested wood pulp in a liquor containing an oxidant and a large excess of alkaline compound for a period of time sufficient to effect a removal of a large proportion of the non-alpha cellulose constituents of such pulp.

12. In the production of fiber characterized by its high alpha cellulose content, that step which comprises treating an alkaline digested wood pulp at about room temperature and under atmospheric pressure in a liquor containing an oxidant and a large excess of alkaline compound for a period of time sufficient to effect a removal of a large proportion of the non-alpha cellulose constituents of such pulp.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.

strength and tear, the fiber produced by the practice of my invention from kraft pulp may be employed instead of rag fiber in the manufacture of high grade bond and ledger papers, and is a suitable substitute for cotton fiber in the manufacture of cellulose derivatives such as the cellulose esters and the cellulose ethers, or for any other purpose where a substantially pure cellulose fiber is required.

I present herein a table illustrating the change in characteristics of a usual commercial kraft stock which has been refined or treated as hereinbefore described.

	Kraft stock	Refined stock (before bleaching)
Strength	155-160	135-145
Tear	240-260	300-330
Pentosan	6-10	2-4
Alpha cellulose	89-90	93-97
Color	Brown	Tan or yellowish.

From the foregoing table, which represents average figures obtained in testing different batches of pulp, it is to be observed that the tear test of the initial raw pulp has undergone a decided increase, while the strength of such pulp has been preserved substantially in entirety. Moreover, it is to be noted that the initial raw pulp, which was relatively low in less-resistant beta and gamma cellulose but high in pentosans (which characteristics are doubtless to be ascribed to the alkaline digestion of the raw cellulosic material in producing such pulp), has been converted into a refined stock, notably high in alpha cellulose content, due to a material reduction in its pentosan content.

Before concluding, it may be stated that, while I have confined myself to a description of the purification of kraft pulp, I have also discovered that a long fibered soda pulp approximating the strength of kraft, i. e., a pulp produced from the digestion of raw cellulosic material, such as spruce and pine, may be treated by the process herein described to produce a high alpha cellulose substantially like that produced from a kraft stock. The various conditions under which the treatment is carried out may vary somewhat, depending upon the characteristics of the soda pulp undergoing treatment.

I do not herein claim the subject matter of application Serial No. 716,153, filed May 27, 1924, by Milton O. Schur and myself wherein a wood pulp is bleached in a liquor which is maintained alkaline during the bleaching operation. In the process of that application the alkaline content of the liquor while sufficient to maintain an alkaline condition of the mass during the entire bleaching operation, is quite subordinate to its bleach content, whereas in the process of the present application the alkaline content of the treating liquor is considerably in excess of its bleach content and the wood pulp undergoing treatment is one derived more particularly from an alkaline digestion of wood chips.

Having thus described this invention, it should be obvious that various changes might be resorted to in the procedure under which the process is carried out, but without departing from the spirit or scope of the appended claims.

What I claim is:

1. In the production of fiber characterized by its high alpha or resistant cellulose content, a step which comprises treating chemical wood pulp in a liquor containing both an oxidizing agent and an excess of alkaline compound for a period of time sufficient to effect a removal of a large proportion of the non-alpha cellulose constituents contained in such pulp.

2. In the production of fiber characterized by its high alpha or resistant cellulose content, a step which comprises treating at a relatively low temperature with a liquor containing bleach and a large excess of caustic soda, pulp derived from an alkaline digestion of wood chips, said caustic soda preponderating in amount and being present in amount sufficient to react with a large proportion of the non-alpha cellulose constituents contained in such pulp but insufficient to effect a substantial mercerization of the alpha cellulose content thereof.

3. In the production of a white fiber characterized by its high alpha or resistant cellulose content, a step of treating pulp derived from an alkaline digestion of wood chips, which process comprises treating such pulp at a stock consistency of about 5% to 6% and at about room temperature for about five or six hours, with a liquor containing 5% to 20% of 35% bleach ($CaOCl_2$) and about 100% to 175% caustic soda, based on the weight of dry pulp.

4. In the production of a fiber characterized by its high alpha or resistant cellulose content, a process which comprises treating alkaline digested wood pulp in a liquor containing bleach and a large excess of caustic soda, for a period of time sufficient to effect the removal of a large proportion of the non-alpha cellulose constituents contained in such pulp, washing the treated pulp, and bleaching the washed product.

5. In the production of a white fiber characterized by its high alpha or resistant cellulose content, a process which comprises treating alkaline digested wood pulp in a liquor containing bleach and a large excess of caustic soda, for a period of time sufficient to effect the removal of the non-alpha cellulose constituents contained in such pulp, washing the treated pulp, bleaching the washed product, and superbleaching the bleached pulp.

6. In the production of a white fiber characterized by its high alpha or resistant cellulose content, a process of treating pulp de-