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PROCESS FOR THE PRODUCTION OF HIGH-ALPHA CELLULOSE FIBER FOR THE MANUFACTURE OF CELLULOSE DERIVATIVES

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This invention relates to the production of fiber containing a high percentage of alpha or resistant cellulose from raw or unbleached pulp, and has for its principal object to produce a white high alpha cellulose fiber which is especially qualified or suitable as a substitute for cotton or cotton linters in the preparation of cellulose esters and other cellulose derivatives, and in the manufacture of cellulose products such as artificial silk, and as a "new rag" substitute in the manufacture of high grade writing papers.

In certain other applications, I have disclosed processes which involve the treatment of raw or unbleached pulp with an alkaline liquor to remove non-alpha cellulose constituents contained therein, followed by a bleaching operation, for the production of a white high alpha cellulose fiber. The alkaline treatment, i. e., the treatment of the raw pulp with an alkaline liquor, moreover, may, in certain instances, be supplemented by a pretreatment of such pulp with a chlorinating or oxidizing liquor, thereby effecting the removal of certain non-alpha cellulose constituents and conditioning such pulp for subsequent alkaline treatment by rendering such non-alpha cellulose constituents more susceptible to removal by the alkaline treatment. The alkaline treatment may be carried out, as desired, either at an elevated temperature, in which case a relatively dilute solution of alkaline purifying compounds is employed, or at a relatively low temperature, (say, room temperature) in which case the concentration of alkaline compounds in the treating liquor is relatively high.

In either case, however, whether the treatment be effected at relatively high or at relatively low temperature, the alkaline treatment, which effects a refinement or purification of the fiber, is followed by a bleaching operation (and then a superbleaching operation, if desired) which, unless carried out under certain conditions and carefully controlled, may affect the alpha cellulose content in the purified fiber secured by previous treatment. Even when the bleaching operation is carried out under the desired conditions, and is controlled, however, a certain small amount of oxycelluloses is present in the bleached high alpha cellulose product. The amount of oxycelluloses present in the final bleached product (assuming that a product having a certain whiteness is desired) is necessarily dependent upon the characteristics of the raw or unbleached pulp employed as a raw material or upon the extent of purification or refinement effected by the treatments precedent to the bleaching operation, or, in other words, upon the characteristics of the fiber before the bleaching operation is effected. Thus a less purified fiber may require more than the moderate amount of bleach required in the bleaching operation of a highly purified fiber to yield the white color of the product desired. In such case, however, the alpha cellulose content, the copper number, and the general papermaking characteristics of the final product are affected. Thus the reduction in the alpha cellulose content and the increase in copper number (owing to increased oxycellulose content of the product) primarily signifies that the fiber has been impaired in value and is less adapted for conversion into cellulose derivatives, as, for example, into viscose, and that the general papermaking characteristics of the pulp have also been impaired.

With the foregoing in view, it might be inferred that a modified procedure which comprises the purification of a bleached pulp, rather than an unbleached pulp, by treatment with an alkaline liquor to produce a white high alpha cellulose fiber having the characteristics desired, should be adopted. I have found, however, that if a bleached pulp is treated with an alkaline liquor, it is impossible to obtain a fiber which has high strength, high tear test, and the desired alpha cellulose content. For instance, in working with a bleached sulphite pulp containing approximately 85% alpha cellulose and having a color of 100 to 102, I found that by an alkaline treatment of such pulp with a liquor containing about 6% to 8% caustic soda, based on pulp, at 180° F. to 212° F., for a sufficient length of time i. e., until an equilibrium in the treatment had been reached, a product was obtained which had an alpha

cellulose content of about 91% and possessed relatively low strength and tear test. Moreover, the yield obtained by such treatment was about 75%, based on the initial raw, bleached sulphite stock, as compared to the 85% yield which is usually obtained by an alkaline treatment of an ordinary unbleached sulphite pulp. The conclusion which may be drawn from this result is that a bleached pulp is very susceptible to injury by an alkaline liquor, and results in a drastic reaction upon the fiber and in a material reduction of its strength and tear test. Principally because of this fact, the process which I employ for the manufacture of a white high alpha cellulose comprehends the alkaline treatment of an unbleached pulp rather than bleached pulp, and a bleaching operation which is subsequent to the purifying alkaline treatment.

I have discovered, however, that the alpha cellulose content and the qualities desired in the white high alpha cellulose fiber produced by the treatment of an unbleached pulp may be improved or enhanced by an alkaline treatment: that is to say that, although the alkaline treatment of a bleached, raw pulp is accompanied by injurious effects on the fiber, a white, purified or high alpha cellulose stock derived from an unbleached raw stock which has been subjected to an alkaline treatment and to a subsequent bleaching operation, may be improved in quality by suitable alkaline treatment. The final fiber thus produced has a very high alpha cellulose content, and a very low copper number, possesses superior papermaking qualities, and is hence particularly adapted for conversion into cellulose esters and other cellulose derivatives, and is also suitable for use as a "new rag" substitute in the manufacture of high grade bond and ledger papers.

The production of the fiber herein desired may be effected from raw or unbleached pulp, by a process comprising various combinations or sequences of treatments. Thus the initial raw stock may, after a preliminary conditioning treatment with an oxidizing or chlorinating liquor, be subjected to an alkaline treatment at an elevated temperature, then bleached, and finally treated with a hot alkaline liquor to yield the desired product.

Another sequence may be the initial conditioning of the unbleached pulp, followed successively by an alkaline treatment at an elevated temperature, a bleaching operation, and an alkaline treatment at relatively low temperature. A further sequence may comprise the initial conditioning treatment of the unbleached raw stock, successively followed by a "cold" alkaline treatment, a bleaching operation, and a hot alkaline treatment, i. e., a treatment at relatively elevated temperatures. Or the sequence of treatments may be the conditioning of an unbleached stock, followed by a treatment with an alkaline liquor at relatively low temperatures (i. e., a "cold" treatment), a bleaching treatment, and a treatment with an alkaline liquor at relatively low temperature.

In each sequence of treatments, the initial or pre-treatment of the raw pulp with a chlorinating or oxidizing liquor which conditions the fiber for purification with an alkaline liquor, may, or may not be employed, depending upon the quality or type of unbleached fiber utilized as a raw material. Thus in certain instances, when the raw pulp is of a "refractory" nature or contains a high percentage of ligneous matter, less-resistant celluloses, and other non-alpha cellulose constituents, as a result of the undercooking of the wood chips or other raw cellulosic material, or because of the quality of such raw material, such pre-treatment of the raw pulp is preferably employed. In such instances, the unbleached stock is treated at about room temperature at a total stock density of about 5%, in a liquor containing about 2% chlorine, or about 2% to 5% lime bleach, based on the weight of dry fiber. The pre-treatment requires a relatively short period of time (about twenty minutes to one hour) and is in no sense a bleaching operation, for the pulp is not whitened thereby. The liquor conditions the pulp for subsequent alkaline treatment, resulting in the solution of certain non-alpha cellulose impurities, or the formation of reaction products which are subsequently dissolved during the treatment with an alkaline liquor. The stock is then washed free of the treating liquor and may be purified by any one of a series of treatments which will presently be described as specific examples.

Example No. 1.—Hot alkaline treatment—Bleaching treatment—Hot alkaline treatment

Unbleached sulphite stock (pretreated, if desired) is treated at a consistency of about 10% in an alkaline liquor containing about 6% caustic soda, based on the weight of stock, or a suitable amount of other equivalent alkaline purifying agent, such as sodium sulphide, at an elevated temperature (180° F. to 212° F.) for about six hours. The alkaline liquor reacts with the non-alpha cellulose constituents contained in the pulp, forming soluble reaction products and thereby purifying the pulp and raising its alpha cellulose content. The purified pulp is washed free of alkaline liquor and entrained products of reaction, and is then bleached at a stock consistency of about 10% with about 5% to 6% lime or sodium bleach, resulting in a pure white product containing 94% alpha cellulose and possessing a copper number of about 2.2. This product is high alpha cellulose fiber. This high alpha cellulose fiber is re-treated at a total stock consistency of about

10% and at an elevated temperature (about 180° F. to 212° F.), with a relatively dilute alkaline liquor containing, say, about 1% to 4% caustic soda, based on the dry weight of pulp.

The treatment is continued for a sufficient period of time to yield a final product having the desired characteristics, or until the oxycelluloses formed in the bleaching operation have been removed and the copper number sufficiently lowered and its alpha cellulose content increased thereby. This treatment may require three to four hours, depending upon the characteristics of the bleached "alpha" stock undergoing treatment. The alkaline treated bleached alpha fiber is washed and is then preferably neutralized in the cold to slight acidity with a weak acid, such as, for example, sulphurous acid solution, resulting in a very white product having an alpha cellulose content of about 95% to 96% and a color of about 100 to 102. The copper number, moreover, has been reduced, testing about 1 to 1.5, and the pentosan content has also been somewhat lowered by the final alkaline treatment. The final fiber is of extremely soft and velvety texture, closely simulating "new rag" stock, and yields solutions of lower viscosity than the bleached and partially purified fiber from which it is prepared, and of materially lower viscosity than the usual or standard cellulosic fiber, such as cotton, sulphite, soda, or kraft. Such change in viscosity is very desirable when solutions of cellulose or cellulose derivatives are employed for purposes where a high concentration and low viscosity of solution is desired, and where the concentration to be obtained is limited by the viscosity of solution thereby produced. These favorable changes in characteristics of bleached alpha fiber have been effected with a yield of about 95% to 98% of the bleached alpha fiber and without adversely affecting its strength and tear test, these values remaining about equal to the corresponding values of the bleached alpha fiber prior to the alkaline treatment.

Example No. 2.—Hot alkaline treatment—Bleach—Cold alkaline treatment

The procedure in this example is similar to that followed in Example 1, with the exception of the final alkaline treatment, which is effected with a stronger alkaline liquor at relatively low temperature, say, approximately room temperature. Thus, by treating a bleached alpha cellulose fiber at a total stock concentration of about 5% in a liquor containing about 25% to 50% caustic soda based on the dry weight of fiber (corresponding to a solution containing 1.25% to 2.50% caustic soda equivalent), for about one hour, a fiber is produced having characteristics approximating those possessed by the fiber produced in accordance with the treatment outlined in Example 1. The pentosan content, moreover, is reduced to a somewhat greater extent by the treatment specified in the present example.

Example No. 3.—Cold alkaline treatment—Bleach—Hot alkaline treatment

Unbleached sulphite fiber (conditioned, if desired) is treated with a relatively concentrated alkaline liquor at relatively low temperature. This treatment is carried out, for example, at a 5% stock concentration, in a liquor containing about 50% to 100% caustic soda, based on the dry weight of fiber. At a treating temperature of about 30° C., the treatment requires about one hour. The purified stock produced by such treatment contains about 94% alpha cellulose and possesses the desired papermaking properties. The stock is then bleached with the usual moderate amount of bleach, then washed, and is finally treated at an elevated temperature with a dilute alkaline liquor under the conditions of final alkaline treatment set forth in Example 1. The product obtained is substantially similar to the products produced in the other example.

Example No. 4.—Cold alkaline treatment—Bleach—Cold alkaline treatment

In this example, the sequence of treatments includes no hot alkaline treatment of the fiber. Thus, the first alkaline treatment is effected at relatively low temperature, under similar conditions to the first alkaline treatment specified in Example 3. The purified fiber is then bleached and finally treated with a relatively strong alkaline liquor, under the condition of final alkaline treatment set forth in Example 2, and resulting in a similar product.

The raw material employed in the examples herein given, was a raw or unbleached sulphite pulp. Various other pulps, however, such as kraft, soda pulp, or pulps produced by various modifications of the sulphite process, may be treated by the process herein set forth. Moreover, the conditions of treatment are given by way of illustration only, and are subject to modification and variation. Thus, the bleaching treatment may be effected in an alkaline bleach liquor, and a superbleaching of the final product herein produced with a chlorine solution may be practiced, if desired. In the treatment of pulps having different characteristics, the concentration of alkaline purifying compounds, bleach, and other reagents used in the various treating steps may be modified to produce the results desired. For example, in certain cases the last alkaline treatment may be effected on the purified pulp which has been prepared under certain modified conditions, so that as a consequence it contains only about 92% alpha cellulose, as compared to a usual alpha cellulose content

of about 94%. In other words, the first alkaline treatment may be carried out in a liquor of a strength lower than usual, thus resulting in a less effective purification of the pulp,—the increase in alpha cellulose content of the pulp to a product having the desired characteristics being effected in the final alkaline treatment on a pulp which has been previously purified to an alpha cellulose content of at least 90%. The various treatments herein proposed are all preferably controlled to yield a final product having approximately similar characteristics, with a yield of such final product from the initial raw pulp as high as is consistent with optimum characteristics.

In the initial and final alkaline treatment, the caustic soda which is employed as the alkaline purifying agent may be replaced in part or in entirety by an equivalent amount of one or a mixture of other suitable purifying reagents, such as sodium carbonate, lime (either dolomite or calcite), or sodium sulphide. However, in an alkaline treatment which is carried out at relatively low temperature, the caustic soda may be replaced only by strongly alkaline agents such as sodium sulphide, as I have been unsuccessful in effecting the desired results under such conditions by the use of sodium carbonate or lime.

Having thus described this invention, it should be obvious that it is susceptible of various changes and modifications without departing from its spirit or scope. Hence I do not wish to limit myself to the specific examples herein given.

What I claim is:

1. A process for the production of a white high alpha cellulose fiber having good papermaking qualities and adapted for conversion into cellulose derivatives, which comprises treating raw pulp with an alkaline liquor to remove non-alpha cellulose constituents contained therein, thereby augmenting its alpha cellulose content to produce a high alpha cellulose fiber; bleaching said fiber; and then treating said bleached fiber with an alkaline liquor to remove non-alpha cellulose constituents contained therein due to said bleaching operation, thereby further augmenting its alpha cellulose content, but without injury to its papermaking characteristics.

2. A process for the production of a white high alpha cellulose-containing fiber, which comprises the following sequence of steps, to wit: conditioning raw pulp for purification in an oxidizing liquor; treating said conditioned pulp with an alkaline liquor to effect a removal of non-alpha cellulose constituents contained therein, thereby producing a high alpha cellulose fiber; bleaching said fiber; and retreating said bleached fiber with an alkaline liquor of a strength to cause a solution of oxy-celluloses formed during bleaching but without injury to its papermaking characteristics.

3. A process for the production of a white fiber of high alpha cellulose content, which comprises treating a raw wood pulp with an alkaline liquor to dissolve non-alpha cellulose constituents therefrom, thereby augmenting its alpha cellulose content; bleaching said pulp; and then treating the bleached pulp with an alkaline liquor containing less than 2.5% caustic soda equivalent to dissolve oxycelluoses formed therein during beaching, thereby further augmenting its alpha cellulose content.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.