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CELLULOSE PURIFICATION

Clifford I. Haney, Greenwich, Conn., and Mervin E. Martin and Troy Monroe Andrews, Cumberland, Md., assignors to Celanese Corporation of America, a corporation of Delaware

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This invention relates to the treatment of wood pulp cellulose and relates more particularly to the treatment of unbleached wood pulp produced by the sulfite process to render the wood pulp suitable for the manufacture of cellulose acetate or other organic derivatives of cellulose or of regenerated cellulose by the viscose or other methods.

Various processes are employed for the separation of cellulosic materials from raw materials such as wood. Some processes yield products which may be satisfactorily employed in many applications, for example, paper-making or in the manufacture of regenerated cellulose materials by the viscose process. Of these processes, the sulfite process yields a wood pulp which has been employed in the preparation of organic derivatives of cellulose. However, sulfite wood pulp has been found to be not wholly satisfactory for conversion into organic derivatives of cellulose since such wood pulp yields cellulose derivatives which are not completely soluble in the usual solvents and possess an undesirable degree of color and form solutions that are generally hazy and difficult to filter so that the derivatives of cellulose are quite unsuitable for many applications, such as the manufacture of filaments, yarns and films. Wood pulps made by other processes are likewise generally subject to the same defects.

It is, therefore, an important object of our invention to provide a simple, efficient and economical process for the treatment of sulfite wood pulp to improve the properties thereof.

Another object of our invention is to provide a novel method for the treatment of wood pulp produced by the sulfite process to render the same suitable for conversion into organic derivatives of cellulose possessing highly desirable characteristics including excellent color and complete solubility in the usual solvents employed.

Other objects of our invention will appear from the following detailed description.

We have now found that sulfite wood pulp which is eminently suitable for conversion into organic derivatives of cellulose by suitable treatment, may be obtained by subjecting the wood pulp to a multi-stage purification. In accordance with the process of our invention, commercial unbleached sulfite wood pulp with or without a prior chlorination treatment is treated with an aqueous alkali solution, washed, chlorinated with chlorine in aqueous solution, and washed again, this cycle being repeated for from 2 to 3 times, ending either with the chlorination step or the alkali treatment step. Preferably, the pulp

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is then given a bleaching treatment with a solution of a hypochlorite, such as sodium hypochlorite.

Sulfite wood pulp treated as above, upon being converted in any known manner to organic derivatives of cellulose, yields highly satisfactory products, particularly organic esters of cellulose, such as cellulose acetate. While the initial treatment of the pulp may comprise a chlorine treatment rather than an alkali treatment as described, when the initial treatment is with aqueous alkali, we have found that the chlorine consumption in the later stages may be substantially reduced, thus effecting the desired purification with only a limited chlorine consumption which renders the process increasingly economical when the cost of chlorine is high. Another advantage of our process resides in the fact that the multiple operations may be carried out advantageously at normal, atmospheric pressure. Super-atmospheric conditions are not necessary and this important factor renders continuous operation simple and quite feasible in open equipment which eliminates excessive cost and the numerous disadvantages of operating under pressure.

In carrying out our novel process for the treatment of sulfite wood pulp, the latter, usually produced in sheet form, is first disintegrated in any convenient manner. Mechanical shredding may be employed and the shredded pulp added to water or the pulp may be digested with water until it is reduced to a slurry of substantially uniform consistency. The pulp is then separated from the water and washed. After washing, with or without a prior chlorination treatment, the pulp is then entered into a dilute aqueous alkali solution, for example, an aqueous solution of sodium hydroxide of a concentration of from 1 to 10% by weight. The amount of pulp added to the solution should be sufficient to form a slurry of which the pulp comprises about 2 to 10% on the weight of the dilute alkali solution. The pulp is then digested for from 10 to 60 minutes at a temperature of from 75 to about 105° C. When the quantity of pulp present is on the order of 8% on the weight of the solution, optimum results are obtained using an aqueous sodium hydroxide solution of about 7% concentration at about 90 to 103° C., the time of treatment being about one hour.

Following the alkali treatment, the pulp is treated with an aqueous solution of chlorine. When the pulp added to the solution forms a slurry containing from 2 to 10% by weight of the pulp, the chlorine may be present in said solu-

tion in an amount varying from 0.5 to 4.0% by weight of the pulp. The chlorine treatment may be carried out for from 30 to 120 minutes and the chlorine consumption may vary from 0.5 to 3.9% on the weight of the pulp present. Preferably, sufficient chlorine solution is employed so that the pulp comprises about 2.5% on the weight of said chlorine solution, and the chlorine treatment is continued for such period that a chlorine consumption of about 0.5% to 2%, say 1.25%, depending upon the purity of the pulp, is effected. After the chlorination is completed the pulp is worked and treated with a solution containing sufficient alkali to give an excess of the order of 0.5 to 1%, the pulp being maintained in the solution for from 20 to 120 minutes at a temperature of about 100° C. At the end of this treatment the pulp is washed.

The pulp is again chlorinated for from 30 to 120 minutes with from 0.5 to 1.0% available chlorine, and washed. Following the chlorine treatment, the pulp is subjected to an alkali boil for from 20 to 120 minutes with relatively dilute sodium hydroxide, i. e. about 0.5 to 1.5% and preferably about 1%, the amount of dilute alkali solution present is such that the pulp comprises from 2 to 10% by weight of the solutions.

The pulp is washed and then is bleached with a solution of hypochlorite, such as sodium hypochlorite containing about 0.5% to 1% available chlorine for from 60 to 300 minutes, or until about 50 to 95% of the available chlorine is consumed. Following this bleach, the pulp is washed and then may either be given a treatment with a dilute solution of acid, such as acetic acid or hydrochloric acid, or may be heated with an aqueous solution of sodium hydroxide of about 1 to 10% concentration, and preferably about 7%, for about an hour at a temperature of 75 to 103° C. After being washed and dried, the purified pulp may then be esterified or etherified in any convenient manner and the purified cellulose yields organic derivatives of cellulose of excellent characteristics.

In carrying out our novel process, we have found that optimum results may be achieved by purifying the pulp in a continuous manner and cycling the aqueous alkali solution employed through more than one stage of the process. The fresh, relatively concentrated alkali solution may be circulated through the process in a direction countercurrent to the pulp. Thus, for example, we have found that it is preferable to employ the aqueous alkali solution first for the last aqueous alkaline treatment of the wood pulp and, after separating the treated pulp therefrom, the solution may then be employed to give the crude pulp entering the system the initial alkaline treatment heretofore described. In this way, the final alkaline treatment is carried out with fresh, uncontaminated aqueous alkali while the initial treatment of the crude pulp is effected with a solution of lesser purity. This counter-current cycling of the relatively concentrated aqueous alkali solution renders the purification of the pulp not only efficient but highly economical as well. If desired, the spent dilute alkali solution resulting after any treatment may be fortified by the addition of alkali to bring it up to the original strength for reuse, or spent alkali solution of greater concentration may be diluted for use in the more dilute alkali treatment.

For the several intermediate aqueous alkaline treatments wherein relatively dilute solutions are employed for treating the pulp, it is most advantageous to employ the aqueous washings which result on washing the pulp following treatment with the relatively concentrated aqueous alkaline solutions. By suitably adjusting the quantity of wash water employed in washing the pulp, a relatively dilute aqueous alkaline solution may be obtained which is of the desired concentration for said intermediate treatments. In this manner, the cycling and recycling of the alkali solution through the several stages in accordance with our process, permits the purification process to proceed in a continuous manner and a highly purified pulp obtained with the consumption of alkali as well as chlorine being maintained at a minimum.

In order further to illustrate our invention but without being limited thereto, the following examples are given:

Example I

Unbleached sulphite wood pulp is disintegrated by mechanical means, cooked with water, and is treated with a 1% aqueous solution of sodium hydroxide, the treatment being carried out at a temperature of 100° C. for 1 hour. After washing, the pulp is chlorinated with 4% aqueous solution of chlorine for 1 hour, the chlorine consumption being about 2.6%. Thereafter, the pulp is treated with an aqueous 7% sodium hydroxide solution for 1 hour at 100° C. It is then given a chlorination treatment with 2% aqueous chlorine solution for 1 hour, the chlorine consumption being 0.42%. A treatment with 7% of sodium hydroxide aqueous solution for 1 hour at 100° C. follows. The pulp is then given a bleaching treatment with 1% sodium hypochlorite aqueous solution for about 3 hours, the consumption of chlorine being about 0.10%, and is treated with a dilute acetic or hydrochloric acid wash to remove any residual alkali.

Example II

Unbleached sulphite wood pulp is disintegrated by mechanical means, cooked with water, and is treated with a 7% aqueous solution of sodium hydroxide, the treatment being carried out at a temperature of 100° C. for one hour. After washing, the pulp is chlorinated with 4% aqueous solution of chlorine for 1 hour, the chlorine consumption being about 2.1%. Thereafter, the pulp is treated with an aqueous 1% sodium hydroxide solution for 1 hour at 100° C. It is then given a chlorination treatment with 2% aqueous chlorine solution for 1 hour, the chlorine consumption being about 0.42%. A treatment with 7% sodium hydroxide aqueous solution for 1 hour at 100° C. follows. Then the pulp is chlorinated with a 0.5% aqueous chlorine solution for 1 hour with a consumption of about 0.09% chlorine. The pulp is then given a bleaching treatment with 1% sodium hypochlorite aqueous solution for about 3 hours, the consumption of chlorine being about 0.09%, and is treated with a dilute acetic or hydrochloric acid wash to remove any residual alkali.

Example III

Unbleached sulphite wood pulp is disintegrated by mechanical means, cooked with water, chlorinated with a 4% aqueous solution of chlorine for 1 hour, the consumption of chlorine being 3.5%. It is then treated with a 7% aqueous solution of sodium hydroxide, the treatment being carried out at a temperature of 100° C. for 1 hour. After washing, the pulp is chlorinated with 2%

aqueous solution of chlorine for 1 hour, the chlorine consumption being about 0.56%. Thereafter, the pulp is treated with an aqueous 1% sodium hydroxide solution for 1 hour at 100° C. It is then given a chlorination treatment with 0.5% of aqueous chlorine solution for 1 hour, the chlorine consumption being about 0.10%. A treatment with 7% sodium hydroxide aqueous solution for 1 hour at 100° C. follows. The pulp is then given a bleaching treatment with 1% sodium hypochlorite aqueous solution for about 3 hours, the consumption of chlorine being about 0.42%, and is treated with a dilute acetic of hydrochloric acid wash to remove any residual alkali.

Example IV

Unbleached sulfite wood pulp is disintegrated and cooked with water and is treated with a 7% aqueous solution of sodium hydroxide recycled from the final alkali treatment. The pulp is present to the extent of about 8% by weight of the alkali solution and the treatment is carried out for one hour at a temperature of 100 to 103° C. The pulp is then washed and treated with a 2% aqueous chlorine solution for 30 minutes. The chlorine consumption is 0.9% by weight of the aqueous chlorine solution during the treatment with sufficient solution being employed so that the wood pulp comprises 2.5%. After washing again, the wood pulp is treated for 30 minutes at 50 to 60° C. with an aqueous solution containing sufficient alkali to give an excess of 0.5% and is washed. The alkali solution employed comprises a portion of the wash water obtained after washing the pulp following the initial alkali treatment, as described. The pulp is again chlorinated, this time for 30 minutes, with 0.5% aqueous chlorine, washed, and then given a boil at 100° C. for one hour in 1% aqueous sodium hydroxide, the amount of solution present at this point is such that the pulp comprises 8% on the weight of the solution. The alkali solution employed in this treatment comprises the wash water obtained when the pulp is washed after the final alkali treatment. After this alkali treatment, the pulp is washed and bleached with an aqueous solution of sodium hypochlorite containing 0.5% available chlorine for 3 hours. The chlorine consumption is about 0.16%. After another washing, the pulp is given a final alkali treatment with fresh 7% aqueous sodium hydroxide solution for one hour at 100 to 105° C. The pulp is then washed and dried, the dried wood pulp obtained exhibiting an excellent white color.

The dried purified pulp prepared in accordance with any of the above examples may be esterified or etherified by processes well-known in the art and cellulose derivatives of very desirable characteristics may be obtained. Examples of cellulose derivatives which may be prepared from the improved wood pulp prepared in accordance with our invention are cellulose esters, such as, for example, cellulose acetate, cellulose nitrate, cellulose propionate, and cellulose butyrate, mixed

esters, such as cellulose acetate-propionate and cellulose acetate-butyrate, and cellulose ethers, such as ethyl cellulose and benzyl cellulose. The pulp may also be used advantageously for the preparation of regenerated cellulose foils, yarn and filaments by the viscose or cuprammonium process.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention, what we desire to secure by Letters Patent is:

1. Process for the treatment of sulfite wood pulp, which comprises forming a slurry comprising 8% by weight of sulfite wood pulp in a 7% aqueous solution of sodium hydroxide, digesting said slurry at a temperature of about 90 to 103° C. for about 1 hour, treating said digested slurry with a 2% aqueous solution of chlorine for about 30 minutes, subjecting said treated slurry to a plurality of treatments with aqueous solutions of sodium hydroxide at a temperature between 50 and 60° C. and aqueous solutions of chlorine, said aqueous solutions of sodium hydroxide and of chlorine being of lower concentration than the aqueous solutions employed in the initial treatment therewith and said aqueous solutions being used for at least 30 minutes, subjecting the so-treated pulp to a bleaching treatment with an aqueous solution of hypochlorite containing 0.5% available chlorine, subjecting said bleached pulp to a treatment with a 7% aqueous solution of sodium hydroxide at a temperature between 75 to 103° C., and then washing and drying said pulp.

2. Process for the treatment of sulfite wood pulp, which comprises forming a slurry comprising 8% by weight of sulfite wood pulp in a 7% aqueous solution of sodium hydroxide, digesting said slurry at a temperature of about 90 to 103° C. for about 1 hour, treating said digested slurry with a 2% aqueous solution of chlorine for about 30 minutes, subjecting said treated slurry to a plurality of treatments with aqueous solutions of sodium hydroxide at a temperature between 50 and 60° C. and aqueous solutions of chlorine, said aqueous solutions of sodium hydroxide and of chlorine being of lower concentration than the aqueous solutions employed in the initial treatment therewith and said aqueous solutions being used for at least 30 minutes, said pulp being washed with water after each sodium hydroxide treatment and after each chlorine treatment, subjecting the so-treated pulp to a bleaching treatment with an aqueous solution of hypochlorite containing 0.5% available chlorine, subjecting said bleached pulp to a treatment with a 7% aqueous solution of sodium hydroxide at a temperature between 75 to 103° C., and then washing and drying said pulp.

CLIFFORD I. HANEY.
MERVIN E. MARTIN.
TROY MONROE ANDREWS.