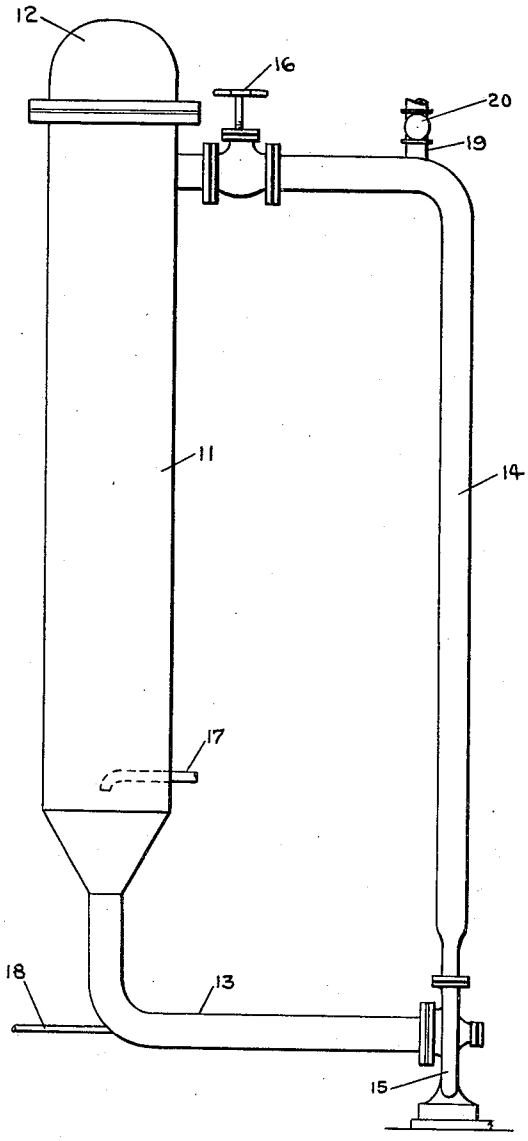
June 26, 1951 M. E. MARTIN ET AL 2,558,054

PURIFICATION OF CELLULOSIC MATERIALS

Filed March 16, 1949



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Patented June 26, 1951 2,558,054

UNITED STATES PATENT OFFICE

2,558,054

PURIFICATION OF CELLULOSIC MATERIALS

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Application March 16, 1949, Serial No. 81,742

2 Claims. (Cl. 8—105)

1

This invention relates to the purification of cellulosic materials, and relates more particularly to the purification of wood pulps.

Cellulosic materials such as wood pulps that are to be employed for the production of cellulose derivatives, such as cellulose esters, must have a high content of alpha cellulose, and must be substantially free from or have a very low content of lignin, pentosans, coloring materials and other impurities. If the cellulosic materials do not meet these requirements, the cellulose derivatives produced therefrom will be hazy and dark, and will not be completely soluble in the solvents normally employed to dissolve them.

It is an important object of this invention to provide a process for purifying cellulosic materials that are to be employed for the production of cellulose derivatives, which will be especially efficient in operation.

A further object of this invention is the provision of an improved process for purifying cellulosic materials, which purified materials will yield cellulose derivatives that are clear and colorless and completely soluble in the solvents normally employed to dissolve them.

Other objects of this invention will be apparent from the following detailed description and claims.

We have now discovered that when cellulosic materials, such as wood pulp produced by the sulfite process, are purified by alternate treatments with chlorine and with an alkali, the pentosan content of the pulp after these treatments may be materially reduced by carrying out at least a part of the alkali treatment at a temperature exceeding about 160° C. Moreover, since the reaction between the impurities in the wood pulp and the alkali proceeds at a much more rapid rate at these elevated temperatures, the time necessary for such treatments may be reduced, which permits important savings to be effected both in steam consumption and in required equipment capacity.

In accordance with the process of our invention, wood pulp produced by the sulfite process is subjected to an initial alkali boil, which may optionally be omitted, and is then chlorinated, and given another alkali boil at a temperature exceeding about 160° C. The wood pulp is also bleached at some point in the process, to brighten the pulp and improve its color.

A preferred embodiment of an apparatus for carrying out the process of our invention is shown in the accompanying drawing, wherein the Figure is a diagrammatic view of a vertical refiner.

In carrying out our novel process, wood pulp is treated for between about 15 and 30 minutes with a sufficient quantity of an aqueous solution of sodium hydroxide having a concentration of

2

between about 0.25 and 1.25% by weight to form a mixture having a consistency of between about 1 and 12% by weight. The treatment is preferably carried out in a closed vessel at a temperature of between about 120 and 135° C. and under a pressure of between about 15 and 35 pounds per square inch. The wood pulp is then separated from the aqueous solution of sodium hydroxide.

Following the alkali treatment and washing, if desired, water is added to the wood pulp to form a mixture having a consistency of between 2.0 and 3.5% by weight, and the mixture is treated for between 30 and 60 minutes with from about 50 to 70% of the chlorine demand of the pulp at this stage as determined by the permanganate number thereof. The chlorination is preferably carried out at a room temperature of from about 10 to 30° C. As has previously been pointed out, the initial alkali treatment may be omitted and the purification of the pulp may begin with its chlorination.

After the chlorination is completed, the pulp is drained and washed with a dilute solution of sodium hydroxide having a concentration of between about 0.02 and 0.20% by weight until the pulp is rendered alkaline. Thereafter, the pulp is treated with a sufficient quantity of an aqueous solution of sodium hydroxide having a concentration of between 0.1 and 5.0% by weight to form a mixture having a consistency of between about 1 and 12% by weight. For the effective removal of the pentosans, the temperature during this alkaline treatment must be at least about 160° C. and may reach about 185° C. The sodium hydroxide need not all be present in the mixture from the very outset, but may be added gradually as treatment proceeds. Since the reaction between the sodium hydroxide and the impurities in the wood pulp proceeds very rapidly at temperatures above about 160° C. the time necessary for this treatment is very short and the maximum consumption of alkali will be reached after about 30 minutes at a temperature of about 160° C. At temperatures above about 170° C., the reaction between the sodium hydroxide and the impurities in the pulp will generally be completed by the time the pulp is brought up to temperature. However, it may be desirable to hold the mixture at the maximum temperature for about 30 minutes even at temperatures above about 170° C. to insure as complete a purification of the pulp as possible.

Instead of subjecting the pulp to a single stage of high temperature alkali treatment, this treatment may be carried out in two or more stages, employing a fresh solution of alkali for each stage. The temperature may be above about 160° C. during each stage of treatment, or the temperature during certain of the stages may be

below this level. In all cases, however, the temperature during at least one stage of treatment must be above about 160° C. to effect a removal of the pentosans from the pulp.

The alternate treatment of the pulp with chlorine and with an alkali will remove the major portion of the impurities therefrom, but will not remove certain coloring materials, which darken the pulp and the products produced therefrom. To remove these coloring materials, the partially purified pulp may be bleached by treatment for about 120 to 240 minutes with a sufficient quantity of an aqueous hypochlorite solution having a concentration of from about 0.25 to 1.0% by weight, of available chlorine based upon the pulp to be bleached, to form a mixture having a consistency of from about 3.5 to 14% by weight. This treatment is preferably carried out at a temperature of from about 25 to 40° C., and during the treatment the pH of the mixture is maintained at about 10.5 by the addition of alkali, if necessary.

Since the high temperature alkali treatment of the pulp renders the coloring materials somewhat more difficult to remove, it may be desirable to carry out part of the bleaching before said treatment. For example, the pulp may be bleached immediately after chlorination by treatment for between about 120 and 240 minutes with a sufficient quantity of an aqueous hypochlorite solution having a concentration of between about 0.25 and 1.0% by weight, of available chlorine based upon the weight of pulp to be bleached, to form a mixture having a consistency of between about 3.5 and 14% by weight. The bleaching is preferably carried out at a temperature of between about 25 and 40° C., and during the bleaching step the pH of the mixture is maintained at about 10.5 by the addition of alkali, if necessary.

Then, after the high temperature alkali treatment, the pulp may be bleached further by treatment for between about 60 and 120 minutes with a sufficient quantity of an aqueous hypochlorite solution having a concentration of between about 0.25 and 0.50% by weight of available chlorine based upon pulp to be bleached, to form a mixture having a consistency of between about 3.5 and 14% by weight. The bleaching is preferably carried out at a temperature of between about 25 and 40° C., and during the bleaching step the pH of the mixture is maintained at about 10.5 by the addition of alkali, if necessary.

The pulp is then washed to remove all traces of the treating agents therefrom, and is finally dried. The alpha cellulose content of the pulp purified in accordance with this invention may reach as high as between about 94 and 95%, and the pentosan content may drop to about 0.7%, or even less. On conversion of the purified pulp to cellulose derivatives, such as cellulose esters, the products obtained are clear and colorless. They are also completely soluble in the solvents, that are normally employed to dissolve cellulose esters, such as acetone, for example.

Among the cellulose derivatives which may be prepared from the wood pulp purified in accordance with this invention are not only the cellulose esters such as cellulose acetate, cellulose propionate, cellulose acetate-proprionate, cellulose acetate-butyrate, but also the celllulose ethers such as ethyl cellulose and benzyl cellulose. The purified wood pulp of this invention may also be employed for the production, by the viscose process, of regenerated cellulose filaments or sheets. In preparing cellulose derivatives, the wood pulp of this invention may be admixed with other cellulosic materials such as cotton linters, or it may be employed alone.

The purification of wood pulp by the process of our invention is best carried out in a vertical refiner of the type shown diagrammatically in the accompanying figure, wherein the reference numeral 11 designates a vertically disposed tank into which the wood pulp may be loaded through a removable cap 12. During the purification, the pulp is circulated from the bottom to the top of the tank 11 through conduits 13 and 14 by means of a centrifugal pump 15, which circulation improves the efficiency of purification and reduces the time necessary therefor. A valve 16 positioned in the conduit 14 controls the rate of circulation of the pulp to prevent overloading of the pump 15. Steam and water, as required, are introduced into the tank 11, to heat and dilute the pulp therein, through a conduit 17, which enters the side of the tank adjacent its base. Chemicals for purification are introduced into the pulp as it flows through the conduit 13 by means of a conduit 18, and are thoroughly mixed with said pulp as it passes through the centrifugal pump 15, insuring a more uniform purification. After purification, the pulp is discharged from the tank 11 by means of the centrifugal pump 15 through a conduit 19 opening into the conduit 14 and provided with a valve 20 to control the flow of the pulp therethrough.

The following examples are given to illustrate this invention further.

Example I

A sufficient quantity of wood pulp produced by the sulfite process is added to a 0.4% by weight aqueous solution of sodium hydroxide to form a mixture having a consistency of 8% by weight. The mixture is heated for 15 minutes to a temperature of 122° C. at a pressure of 15 pounds per square inch in a closed digester. The pulp is drained and added to a sufficient quantity of water to form a mixture having a consistency of 2% by weight. To this mixture are added 1.5 parts by weight of chlorine for each 100 parts by weight of the pulp and chlorination permitted to proceed for 50 minutes at a temperature of 25° C. After chlorination, the pulp is drained and washed alkaline with a 0.2% solution of sodium hydroxide. A sufficient quantity of the pulp is then entered into a 1.3% by weight aqueous solution of sodium hydroxide to form a mixture having a consistency of 6.25% by weight. The mixture is heated in a closed digester to a temperature of 170° C. and held at this temperature for 15 minutes. Even without bleaching the pulp has an alpha cellulose content of 93% and a pentosan content of only 0.6%. The pulp is drained and a sufficient quantity thereof is entered into an aqueous solution of sodium hypochlorite having a concentration of 0.30% by weight of available chlorine based upon the pulp to form a mixture having a consistency of 3.5% by weight. After bleaching for 120 minutes at 35° C., the pulp is washed and dried. When converted to cellulose acetate by conventional methods, the purified pulp produces an ester which is clear and colorless, and completely soluble in acetone.

Example II

A sufficient quantity of wood pulp produced by the sulfite process is added to water to form a mixture having a consistency of 3.5% by weight. To this mixture is added 60% of the chlorine

demand of the pulp and chlorination permitted to proceed for 30 minutes at a temperature of 25° C. After chlorination, the pulp is drained and washed alkaline with a 0.20% solution of sodium hydroxide. A sufficient quantity of the pulp is entered into a 0.9% by weight aqueous solution of sodium hydroxide to form a mixture having a consistency of 6% by weight. The mixture is heated in a closed digester to a temperature of 160° C. and held at this temperature for 15 minutes. Even without bleaching the pulp has an alpha cellulose content of 94.4% and a pentosan content of only 1.1%. The pulp is drained and bleached as set forth in Example I.

Example III

A sufficient quantity of wood pulp which has been chlorinated and washed alkaline as described in Example II is entered into a 0.7% by weight aqueous solution of sodium hydroxide to form a mixture having a consistency of 6.6% by weight. The mixture is heated in a closed digester to a temperature of 170° C. and held at this temperature for 15 minutes. The pulp is drained and a sufficient quantity thereof is entered into an aqueous solution of sodium hypochlorite having a concentration of 0.50% by weight of available chlorine based upon the pulp to form a mixture having a consistency of 3.5% by weight. After bleaching for 120 minutes at 35° C. the pulp is drained and a sufficient quantity thereof is entered into an aqueous solution of sodium hydroxide having a concentration of 0.1% by weight to form a mixture having a consistency of 6% by weight. The mixture is heated in a closed digester to a temperature of 173° C. and maintained at this temperature for 30 minutes. After washing and drying, the purified pulp has an alpha cellulose content of 95.0% and a pentosan content of only 1.0%.

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. In a process for purifying cellulosic materials, the steps which comprise treating said cellulosic materials for between about 15 and 30 minutes at a temperature of between about 120 and 135° C. with a sufficient quantity of sodium hydroxide having a concentration of between about 0.25 and 1.25% by weight to form a mixture having a consistency of between about 1 and 12% by weight, treating said cellulosic materials in the form of an aqueous mixture having a consistency of between about 2.0 and 3.5% by weight with chlorine in an amount of from about 50 to 70% of the chlorine demand of the cellulosic material for between about 30 and 60 minutes at a temperature between about 10 and 30° C., and treating said cellulosic materials for up to about 30 minutes at a temperature between about 160 and 185° C. with a sufficient quantity of an aqueous solution of sodium hydroxide having a concentration of between about 0.1 and 5.0% by weight to form a mixture having a consistency of between about 1 and 12% by weight.

2. In a process for purifying cellulosic materials, the steps which comprise treating said cellulosic materials for between about 15 and 30 minutes at a temperature between about 120 and 135° C. with a sufficient quantity of an aqueous solution of sodium hydroxide having a concentration of between about 0.25 and 1.25% by weight to form a mixture having a consistency of between about 1 and 12% by weight, treating said cellulosic materials in the form of an aqueous mixture having a consistency of between about 2.0 and 3.5% by weight with chlorine in an amount of from about 50 to 70% of the chlorine demand of the cellulosic material for between about 30 and 60 minutes at a temperature between about 10 and 30° C., treating said cellulosic materials for up to about 30 minutes at a temperature between about 160 and 185° C. with a sufficient quantity of an aqueous solution of sodium hydroxide having a concentration of between about 0.1 and 5.0% by weight to form a mixture having a consistency of between about 1 and 12% by weight, and bleaching said cellulosic materials with an aqueous solution of sodium hypochlorite before said cellulosic materials are treated with an aqueous solution of sodium hydroxide at a temperature between about 160 and 185° C.

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