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PAPER AND THE PROCESS OF MAKING CELLULOSE XANTHATE FROM IT

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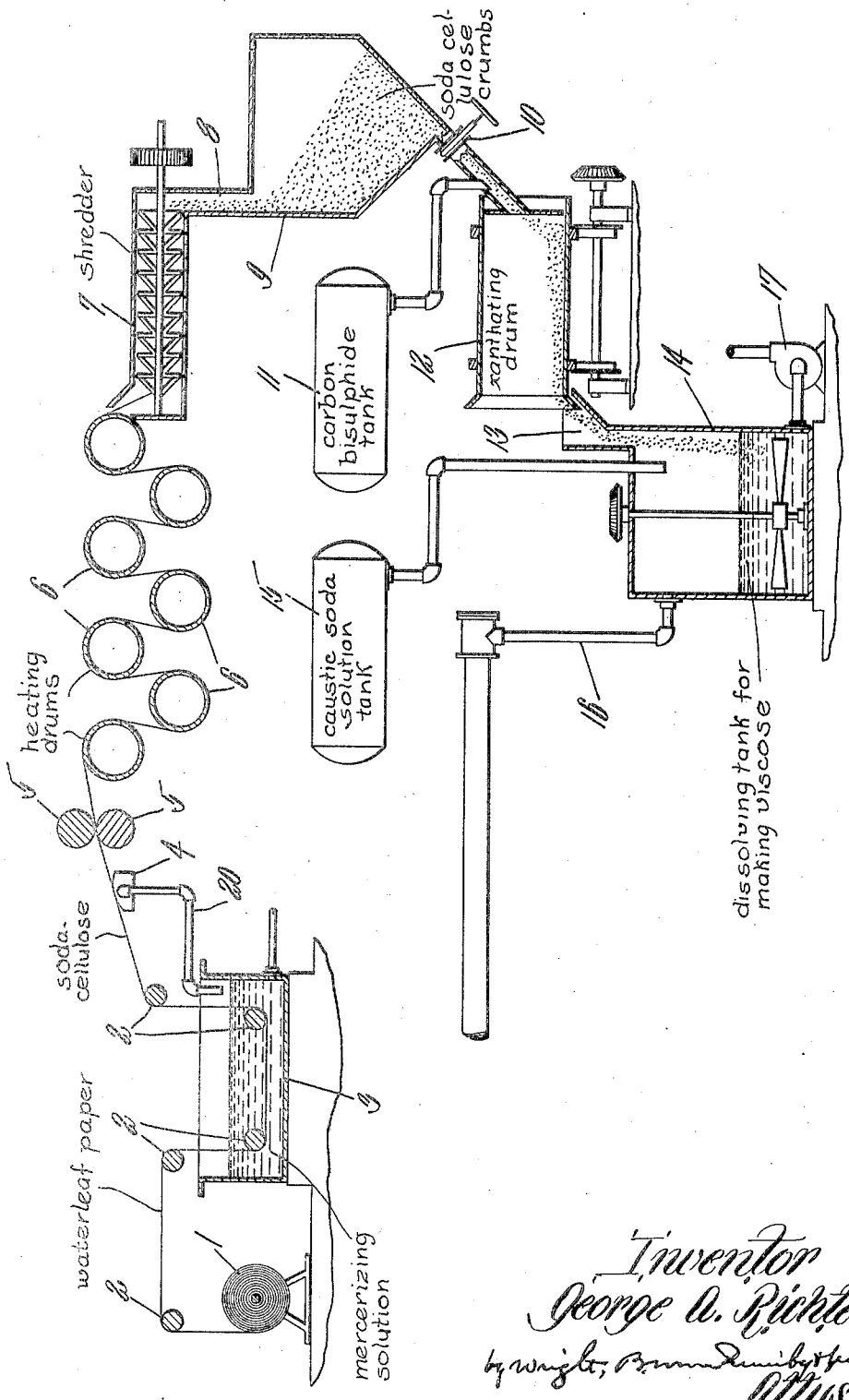

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PAPER AND THE PROCESS OF MAKING CELLULOSE XANTHATE FROM IT

Application filed May 26, 1927. Serial No. 194,506.

In the manufacture of viscose-rayon, it has been the practice to employ as a raw material, cellulose fiber in substantially unbeaten condition and in the form of drier sheets, so called. These sheets are thoroughly soaked with caustic soda solution, then pressed to remove excess solution therefrom, and then disintegrated into a bulky mass, which is aged to effect sufficient depolymerization to undergo substantially complete xanthation.

In my application, Serial No. 196,135, filed June 2, 1927, I have disclosed a process which comprises materially reducing cellulose fiber in size prior to processing as hereinbefore described. Such reduction in fiber size makes possible a much shorter ageing period, and in some cases permits the elimination of ageing altogether. Furthermore, the reduced fiber is substantially uniformly depolymerized and hence xanthates more readily and completely than fiber of natural or original size.

In accordance with the process of the present invention, the reduced or shortened fiber is formed into waterleaf paper or tissue prior to processing, as I have found that the fiber may be handled and treated much more expeditiously and satisfactorily while in such condition. Thus, the reduced or shortened fiber may be run off into a continuous sheet of paper, which may be continuously soaked with caustic soda solution and pressed free of excess solution, and, if necessary, continuously depolymerized while in sheet form, thus making possible the practice of continuous operation where batch operation was heretofore found to be necessary.

In practicing the process of my invention, cellulose fiber of any suitable type for rayon manufacture may be employed for raw material. Thus, I may preferably employ cotton fiber, sulphite fiber, or a refined chemical wood fiber composed essentially of alpha cellulose. Inasmuch as such fiber unless further treated comprises component fibers of widely varying sizes, it has heretofore been necessary to age such fiber until substantially all the large fibers were sufficiently depolymerized or activated for xanthation. Hence, the ageing period was long and the aged product non-uniformly depolymerized, the smaller fibers being depolymerized to a greater extent than the larger fibers, sometimes even being over-depolymerized.

In accordance with my process, the fiber is materially reduced in size, as by beating, but this operation need not effect hydration. The fiber is thereby activated for xanthation, as reduction of fiber size is apparently accompanied by depolymerization. Thus, whereas the original fiber requires a long ageing period—usually from two to four days—to become sufficiently depolymerized for xanthation, the reduced or shortened fiber may be xanthated with little, if any, ageing. The reduced fiber is run off into waterleaf paper, preferably of light weight, as such paper may be processed most effectively. The paper may be passed through a caustic soda solution in continuous fashion, and then between press rolls, which serve to remove excess solution therefrom, after which it may be depolymerized, if necessary, while still in continuous sheet form. The paper may finally be xanthated in sheet form or after disintegration into a bulky mass, as usual. Inasmuch as the fibers of the mass are small and of substantially uniform size and substantially uniformly depolymerized, xanthation takes place quite readily and completely, and the xanthated product dissolves in caustic soda solution to produce a xanthate solution containing only a small amount of unxanthated fiber.

A more complete understanding of my process may be obtained from the following more complete description thereof when considered in conjunction with the accompanying drawing, which represents diagrammatically and conventionally certain apparatus which may be employed.

As a raw material, I may utilize cellulose fiber or a mixture of fibers of any desired origin and suitable for rayon manufacture. The fiber is materially reduced in size from its natural or original condition, but this need not be effected under conditions resulting in hydration, as I am not especially interested in the production of paper having physical characteristics other than those necessary for

handling. Reduction in fiber size may be accomplished as by passing the fiber through a jordan or charging it into a hollander having a beater roll equipped with sharp blades slightly clearing the bed-plate. Jordaning or beating may take place at elevated temperature, this being undesirable in usual papermaking procedure, as it tends to decrease the hydratability of the fiber, and may be continued until a fiber having an average fiber length of, say, 0.7 to 1.3 mm. is reduced to an average fiber length below 0.2 mm.

The shortened fiber is then run off on a paper machine into waterleaf paper or tissue of suitable caliper,—for instance, paper of 20 to 80 pounds basis weight. Paper of low basis weight is preferable, as it may be processed more effectively than paper of high basis weight. If the fiber has not been hydrated to any material extent, its freeness permits paper manufacture to be carried out at a high rate of speed.

As illustrated on the accompanying drawing, the paper thus produced is unwound from a reel 1, and as shown is passed over and under suitable guide rolls 2, through a caustic soda solution bath maintained in a tank 3. The solution may be of a strength and at a temperature to effect a conversion of the fiber into soda cellulose. Thus, a solution of the usual 18% strength and, if desired, containing a certain amount of caustic potash and lithium hydroxide, may be employed at room temperature. The paper is rapidly permeated by the solution, and becomes substantially uniformly converted into soda cellulose. In lieu of passing the paper through one bath of solution as shown, it may be passed through a series of such baths while passing the solution from bath to bath in counter-current flow to the direction of travel of the paper. This procedure produces a purer soda cellulose, as in passing through the successive baths, non-alpha cellulose components such as pentosans, lignin, and other coloring impurities present in the fiber are dissolved therefrom, the concentration of these impurities in solution decreasing from bath to bath toward the last bath, so that when the paper emerges from the last bath, it contains substantially pure solution. This latter procedure is particularly effective when treating an ordinary fiber of wood origin, as it results in a xanthate solution containing a low amount of coloring impurities.

Provision is now made to remove excess solution from the saturated paper. As shown, this is accomplished by passing the paper as it emerges from the solution bath over a suction box 4, and thence between press rolls 5 stationed close to the suction box. The solution thus removed may be reused by passing it from the suction box through the pipe 20 into the tank 3.

At this stage in the process, it may be desirable to depolymerize the fiber somewhat, or to prevent depolymerization. If depolymerization is desired, the paper may be heated somewhat and/or treated with an accelerator of depolymerization. If, on the other hand, depolymerization is to be prevented, the paper may be cooled to a temperature whereat inappreciable depolymerization takes place. The desired end may be attained by passing the paper over a series of supporting rolls 6, which may be constructed similar to paper dryer rolls, and which may be maintained at the desired temperature by circulating a heating or cooling medium therethrough. For instance, the rolls may be maintained at, say, 40° to 50° C., at which temperature depolymerization takes place quite rapidly. After such heating, the paper may be cooled, as by running it over cooling rolls, to the temperature at which it is desired to carry out xanthation. Inasmuch as cooling greatly reduces the rate of depolymerization, upon reaching the desired depolymerized condition of the paper, it may be cooled quickly to prevent further material depolymerization. If desired, air, oxygen, or other depolymerization accelerator, at room or elevated temperature, may be delivered onto the paper while it is supported on the rolls, without circulating a medium through the rolls, or while it is festooned, in case rolls are not used. On the other hand, if depolymerization is to be prevented, the paper may be maintained in contact with the atmosphere for only a very short time, or may be cooled to below room temperature while supported on the rolls 6. If depolymerization is effected by passing the paper over heated dryer rolls, a certain amount of water contained in the paper is evaporated, thereby increasing the concentration of the caustic soda solution in the paper. Hence, in such case, the saturation of the paper with caustic soda solution may be effected with a solution of lower concentration than usual if the proper ratio between caustic soda and fiber is present in the pressed paper. The paper may then be xanthated in continuous sheet form, as by passing it through a xanthating atmosphere, or disintegrated into a bulky mass prior to xanthation, as is usually done. As shown the paper passes into a disintegrator 7, which may be of the usual shredder type and which serves to disintegrate the paper into a fluffy or bulky mass of crumbs of high specific volume,—1000 cc. for instance, weighing about 200 grams. The crumbs may be discharged through a spout 8 into a storage hopper 9, from which they may be withdrawn for xanthation. As indicated, they gravitate through a valved discharge conduit 10 and mingle with carbon bisulphide fed into the conduit in controlled amount from a tank 11, both materials passing into one end of a rotary reacting drum 12, wherein they under-

go reaction under controlled temperature conditions to form cellulose xanthate. The cellulose xanthate is delivered from the other end of the drum into a spout 13 discharging into a dissolving tank 14, whereinto caustic soda solution from a storage tank 15 and water from a supply pipe 16 are fed in requisite amount to form an aqueous solution of the desired concentration. Any unxanthated fiber present in the solution may be separated therefrom by filtration, a pump 17 being shown for withdrawing the solution from the tank 14 for further processing. Inasmuch as the soda cellulose crumbs are composed of small fibers depolymerized approximately to the same degree, substantially complete xanthation takes place quite readily in the drum, and the xanthated fibers dissolve to form a solution containing only a small amount of unxanthated fiber.

Where it is desirable to carry out the processing up to shredding independently of the remainder of the processing, the paper is wound up quite tightly into rolls as it comes from the roll 6 in the desired depolymerized condition. These rolls of paper contain a relatively small amount of air, so that depolymerization does not take place at room temperature to a material extent, even over a number of days.

The processing hereinbefore described may be performed upon several plies of paper at the same time. Thus, several rolls of paper may be unwound simultaneously, and their respective plies passed separately in parallel relation through the solution in the tank 3, whereupon they may be united and passed between the press rolls 5, and subsequently handled as a single sheet. Using relatively thin papers, ready and complete penetration by the solution is secured. Where somewhat heavy papers are used, they may be perforated to aid permeation by the solution, depolymerization, and subsequent shredding.

I do not herein claim broadly the subject matter of my application Serial No. 196,135, filed June 2, 1927, which discloses the advantages of materially reducing cellulose fiber in size prior to conversion into cellulose derivatives such as cellulose xanthate, nor do I herein specifically claim the subject matter of my application Serial No. 122,271, filed July 13, 1926, which discloses the advantages of beating those pulps which undergo disintegration if soaked in unbeaten sheet form in a solution of caustic soda, prior to conversion into cellulose xanthate.

Having thus described this invention, it is obvious to those skilled in the art that change and modification might be made therein without departing from the spirit or scope of the appended claims.

What I claim is:

1. Waterleaf paper especially suited for xanthation, having perforations therein and comprising fiber having an average fiber length below about 0.2 mm.

2. A process which comprises markedly reducing cellulose fiber in size, running such reduced fiber off into waterleaf paper, and converting such paper into cellulose xanthate.

3. A process which comprises markedly reducing cellulose fiber in size, running such reduced fiber off into waterleaf paper, treating such paper with caustic soda solution, and converting the treated paper into cellulose xanthate.

4. A process which comprises reducing cellulose fiber in size, running such reduced fiber off into waterleaf paper, treating such paper with caustic soda solution, disintegrating the treated paper into crumbs, and xanthating the crumbs.

5. A process which comprises beating cellulose fiber until its average fiber length is reduced to below about 0.2 mm., running such beaten fiber off into paper, and converting such paper into cellulose xanthate.

6. A process which comprises treating a continuous sheet of waterleaf paper composed of markedly shortened fibers to form soda cellulose, and continuously depolymerizing the paper in an atmosphere of oxidizing gas while in sheet form.

7. A process which comprises passing a continuous sheet of waterleaf paper composed of markedly shortened fibers through caustic soda solution, continuously removing excess solution therefrom, and continuously depolymerizing such paper in an atmosphere of oxidizing gas while in sheet form.

8. A process which comprises passing a continuous sheet of waterleaf paper composed of markedly shortened fibers through caustic soda solution, continuously removing excess solution therefrom, and continuously depolymerizing such paper in an atmosphere of oxidizing gas under accelerated depolymerizing conditions while in sheet form.

9. A process which comprises passing a continuous sheet of waterleaf paper composed of markedly shortened fibers through caustic soda solution, continuously removing excess solution therefrom, and continuously depolymerizing such paper in a heated atmosphere of oxidizing gas while in sheet form.

10. A process which comprises passing a continuous sheet of waterleaf paper composed of markedly shortened fibers through caustic soda solution, continuously removing excess solution therefrom, and continuously depolymerizing such paper in a heated atmosphere of oxidizing gas and in the presence of an accelerator of depolymerization while in sheet form.

11. A process which comprises passing a continuous sheet of waterleaf paper composed of shortened fibers through a caustic soda

solution, continuously removing excess solution therefrom, continuously depolymerizing such paper while suspended and guided in sheet form, and continuously disintegrating the depolymerized paper into crumbs.

12. A process which comprises passing a continuous sheet of waterleaf paper composed of shortened fibers through a caustic soda solution, continuously removing excess solution therefrom, continuously depolymerizing such paper while suspended and guided in sheet form, continuously disintegrating the depolymerized paper into crumbs, and xanthating the crumbs.

13. A process which comprises passing a continuous sheet of paper composed of markedly shortened fibers through a series of baths of caustic soda solution while passing the solution from bath to bath in counter-current flow to the direction of travel of the paper, continuously removing excess solution therefrom, and converting the paper into cellulose xanthate.

14. A process which comprises passing a plurality of continuous sheets of waterleaf paper composed of markedly shortened fibers separately through a caustic soda solution, uniting said sheets into a single sheet, pressing excess solution from said sheet, and converting said sheet into cellulose xanthate.

15. A step product in the manufacture of cellulose xanthate, consisting of paper whose fibers not only are markedly reduced in size from their natural or original condition but are combined with caustic soda as soda cellulose and are substantially uniformly depolymerized.

16. A step product in the manufacture of cellulose xanthate, consisting of paper comprising substantially uniformly depolymerized fibers combined with caustic soda as soda cellulose and of an average fiber length below about 0.2 mm.

17. A process which comprises beating cellulose pulp to reduce the length of the pulp fibers, forming the beaten pulp into waterleaf paper, saturating such paper with a solution of caustic soda, pressing excess solution from such paper, shredding the paper, and converting the shreds of paper into viscose.

18. A process which comprises beating cellulose pulp to reduce the length of the pulp fibers, forming the beaten pulp into waterleaf paper, and converting such paper into cellulose xanthate.

19. A process which comprises beating cellulose pulp to reduce markedly the length of the pulp fibers, sheeting into waterleaf paper, passing a continuous sheet of such paper through a caustic soda solution of mercerizing strength to form soda cellulose, progressively depolymerizing such fiber in an atmosphere of oxidizing gas while in sheet form, and converting the depolymerized paper into cellulose xanthate.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.