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PROCESS OF MAKING VISCOSE

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This invention relates to a process of making viscose or cellulose xanthate solution involving the admixture and reaction all at once of the various ingredients, namely, the cellulose, caustic soda solution, and liquid carbon bisulphide, necessary for the preparation of viscose or cellulose xanthate solution. Its objective is to condition cellulose pulp in predried condition, e. g., in the form of pulpboard or so-called drier sheets, for the one-step xanthating reaction.

In preparing chemical wood pulp or the like for storage and shipment, it is customary to dewater the pulp from its water-suspended state on a pulp drier, which operates essentially as a papermaking machine and delivers the pulp as a substantially dry board or sheet that can readily be cut into the drier sheets of commerce. Pulp in the form of drier sheets has been and is currently being supplied to the viscose-making industry. In the usual viscose-making process, the sheets are soaked in strong caustic soda solution in a so-called steeping press; the soaked sheets are freed of excess solution in the press and are then disintegrated into crumbs or shreds of soda cellulose; and the crumbs, after suitable ageing, are xanthated while being tumbled in a drum with carbon bisulphide vapor and finally dissolved in dilute caustic soda solution to form viscose. It is thus seen that pulpboard represents a physical form of pulp that lends itself nicely to handling during the initial soaking and pressing steps of the usual viscose-making process.

In making viscose by a one-step xanthating process, the physical form of pulp represented by pulpboard or drier sheets is of no particular value, excepting insofar as such form was previously advantageous for shipment and storage purposes. Indeed, for a one-step xanthating process, it is important that the cellulose fibers be individually suspended in caustic soda solution so that the liquid carbon bisulphide admixed with the suspension can have free access to the fiber surfaces and thus react smoothly and substantially completely with the fibers to yield in a reasonable period of time a viscose or cellulose xanthate solution substantially devoid of unxanthated or incompletely xanthated cellulose fibers or particles.

In accordance with the present invention, predried cellulose pulp, as in the form of pulpboard or drier sheets, is first slushed or disintegrated in sufficient water to yield a relatively dilute or fluent aqueous pulp suspension, namely, one of a fiber content downwards of 6%, whereupon the pulp suspension is thickened to much higher fiber content, for instance, about 20% to 40%, an the thickened wet pulp admixed with strong caustic soda solution to form a suspension in caustic soda solution of a strength or causticity appropriate for the one-step xanthating reaction. To the resulting suspension is added liquid carbon bisulphide in amount suitable for forming the desired viscose or cellulose xanthate solution; and the mixed ingredients are allowed to react to form viscose or cellulose xanthate solution under the appropriate conditions. The process hereof is advantageous in that, although the fibers in pulpboard are interfelted or firmly integrated as a comparatively dense structure, yet the slushing or disintegration of the pulpboard in a comparatively large volume of water, for instance, a volume of water to yield a pulp suspension of about 2% to 4% fiber content, can be readily accomplished in a beater engine, hammermill, or other disintegrator or slushing apparatus. Indeed, the pulp sheets may be reduced or slushed in water in an ordinary high-speed mixing tank, wherein the pulp fibers are not appreciably hydrated, to yield the desired aqueous pulp suspension wherein the pulp fibers are practically completely individualized. By using a large amount of water for slushing the pulpboard, not only is the pulpboard quickly swollen and broken up into fragments, but the aggregated or interfelted fibers are given ample opportunity to become uniformly dispersed or individualized in the large amount of aqueous suspending medium. In other words, in the presence of ample aqueous suspending medium to yield a readily fluent pulp suspension, the aggregated or interfelted fibers can, under the mechanically disruptive forces of a beater engine, hammermill, or other slushing or mixing apparatus, be readily resolved into the individual fibers on account of their freedom of locomotion or dispersion in the aqueous medium. While the initial disintegration of the dried pulp to form a dilute pulp suspension makes necessary a subsequent thickening of the pulp preparatory to its admixture with strong caustic soda solution in order to arrive at a pulp suspension in caustic soda solution of desirably high fiber content, preferably greater than 6%, for making viscose for artificial silk, films, and other ultimate products, yet the thickening of a dilute pulp suspension is a relatively simple and inexpensive matter and, so long as the thickened pulp is kept thoroughly wet, its fibers may be redispersed substantially uniformly and without any difficulty whatever in the diluted caustic soda solution, prefer-

ably of less than about 18% strength, formed by admixing strong caustic soda solution with the thickened wet pulp. When it is intended to disintegrate pulpboard or the like in caustic soda solution to form directly a suspension of greater than 6% fiber content, considerable difficulty is experienced in developing a suspension substantially free from fiber aggregates or clumps, unless the pulpboard is initially cut into chips or small pieces and the chips are soaked or swollen thoroughly with caustic soda solution of mercerizing activity and then diluted with water, as described in Richter application Serial No. 58,539, filed January 10, 1936. The process hereof not only dispenses with the step of cutting pulpboard into chips but enables the desired uniform dispersion or individualizaton of the fibers in plain water or only very mildly alkaline water. Standard equipment is available for the slushing and thickening steps of the process hereof and the cost of operating such equipment is comparatively low. Moreover, the process hereof is advantageous over the usual viscose-making process in that, whereas pulp sheets for the usual viscose-making process must be made to conform to exacting specifications of thickness, compactness, size, etc., it is possible for the pulp producer to prepare and ship the dried pulp for the process hereof in the form of stacked sheets or rolls without regard to such particular specifications. The pulp producer is thus enabled to reduce his costs materially, since the item of cost incident to preparing the special sheets for the usual viscose-making process is considerable.

The pulpboard or drier sheets used in the process hereof may be composed of any suitable chemical wood pulp, such as bleached sulphite pulp or wood pulp refined to supernormal alpha cellulose content; or the predried aggregated cellulose subjected to the process hereof may be cotton in sheet form or in the form of linters. The drier sheets of wood pulp may be supplied to the usual hollander or beater engine together with sufficient water to produce a relative freely fluent fibre suspension, say, one of about 4% to 2% or even lower fiber content. As the sheets are broken down into fragments, the beater roll may be gradually lowered toward the bed-plate to brush out the sheet fragments and complete the liberation or individualization of the pulp fibers. Indeed, the operation of the beater engine may be continued beyond the stage of fiber-individualization to the stage of effecting more or less cutting or reduction of the pulp fibers and also hydration thereof. If desired, a small amount of caustic soda, say, in amount to produce a solution of less than 1% strength, may be added to the beater water for the purpose of promoting disintegration of the drier sheets and the individualization of the pulp fibers. If desired, the slushing of the pulp sheets in water may be effected in a disintegrator or mixing apparatus other than a beater engine, which latter is cited simply for the purpose of illustration. The water in which slushing of the pulp sheets is effected may vary in its temperature, depending upon the particular kind of pulp being slushed. Thus, warm water promotes the disintegration of some kinds of pulp, whereas cold water favors the disintegration of other kinds of pulp. Low-temperature slushing water induces swelling of the pulp fibers suspended in such water and uniform absorption of the caustic soda solution by the fibers of the subsequently thickened suspension. The use of cold water for slushing the sheets may hence be preferable, especially when thickening of the resulting pulp suspension need not be carried to the highest pulp to water ratios. In this latter connection, it might be noted that the use of cold water as the slushing medium renders somewhat more difficult the thickening of the slushed pulp to the same high degree attainable with warm water.

The dilute aqueous pulp suspension devoid of fiber clumps or aggregates thus produced in the beater engine or equivalent disintegrator or mixer is thickened to much higher fiber content, for instance, to a fiber content of about 20% to 40%. The thickening of the dilute pulp suspension may be accomplished by the usual rotary thickener, on whose foraminous periphery the thickened pulp layer may be more or less pressed before it is removed by a doctor blade and/or whose hollow interior may be under appropriate suction; or the dilute pulp suspension may be thickened to the desired higher consistency in a centrifuge or in any other suitable manner. The thickened or wet pulp may then be put into a suitable mixing and reacting vessel along with strong caustic soda solution, say, liquid caustic soda of commerce of 50% strength. Such strong caustic soda solution may be used in volume to yield a suspending solution of a strength of, say, about 6% to 15%, taking into consideration the dilution of such solution effected by the water content of the wet or thickened pulp. The fiber content of the resulting dilute suspension may also fall within a range of, say, about 6% to 15%. If, as is usually desired for artificial silk and film manufacture, the resulting viscose is to contain approximately equal proportions each of cellulose and caustic soda, say, about 7% each, based on the weight of the viscose, the fluent caustic soda solution admixed with the thickened pulp is of a strength approximating the fiber content of the thickened or wet pulp; or a stronger caustic soda solution may be employed together with sufficient extraneous diluting water to yield when admixed with the thickened pulp the desired fiber suspension of approximately the same fiber content and causticity. In some instances, it may be desirable to add the desired amount of caustic soda in the form of a sufficiently strong caustic soda solution to the thickened pulp to produce a solution of about 18% or greater strength as the suspension medium for the fibers and, after such caustic soda solution has been kept in contact with the fibers for a while, to add an amount of water to reduce the suspension or mixture of pulp and caustic soda solution to the consistency or composition desired for xanthation. For some pulps, such procedure is advantageous in that the strong caustic soda solution brought in contact with the pulp is more effective in swelling the pulp fibers and conditioning them for xanthation than the weaker caustic soda solution in the presence of which xanthation is actually effected.

To the resulting fiber suspension in caustic soda solution is then added the amount of liquid carbon bisulphide necessary for forming viscose or cellulose xanthate solution of the desired character. The amount of liquid carbon bisulphide may vary, as indicated in Richter and Lovering application Serial No. 153,688, filed July 14, 1937, depending upon such factors as the characteristics of the particular fiber employed, the causticity of the fiber suspension, the temperature maintained during the xanthating reaction, etc. The presence in the liquid carbon bisulphide of an organic liquid serving to impart thereto a

specific gravity approximating that of the caustic soda solution in which the fiber is suspended promotes the desired xanthating reaction, as disclosed in the aforementioned Richter and Lovering application; and, as also described in that application, if the organic liquid is immiscible with caustic soda solution, it may be desirable to add a small amount of soap or other emulsifying agent to the mixed ingredients.

The process hereof is not restricted to the use of predried pulp in the form of pulpboard or drier sheets. Thus, it is applicable to pulp predried in bulk to form relatively dense cakes or masses which, although resistant to yielding directly thick aqueous fiber suspensions of the desired smoothness or freedom from fiber clumps, may be slushed easily in water to form comparatively fluent pulp suspensions devoid of fiber aggregates. The use of small amounts of wetting agents of the nature of the water-soluble soaps in the slushing water facilitates the opening up of the fiber aggregates or clumps constituting the predried pulp and the desired substantially complete individualization of the pulp fibers in the water. The use of soap or other wetting agents in the slushing step of the process may prove more effective in individualizing the pulp fibers than when such agents are added in the later stages of the process, wherein the strong caustic soda solution may decompose the soaps or other wetting agents or otherwise impair their effectiveness. The comparatively fluent or dilute pulp suspension initially prepared from the predried pulp may be thickened to various consistencies for mixing or redilution with strong caustic soda solution, provided that thickening or dewatering of the pulp suspension is not carried so far that the fibers of the thickened pulp again become strongly integrated or resistant to suspension. Generally speaking, the thickening or dewatering of the dilute pulp suspension is carried to a stage whereat, although the thickened pulp is essentially non-fluent and cannot be pumped or otherwise handled like water, it is still very wet and has a water to pulp ratio distinctly greater than 1 to 1, that is, contains water in amount distinctly greater than 100%, based on the dry weight of the pulp. The fiber suspension produced by admixing the thickened pulp with caustic soda solution, although sufficiently mobile and smooth to enable the desired uniform mixing with the liquid carbon bisulphide, is of a thick, paste-like consistency at fiber contents upwards of 6%.

The process hereof has noteworthy advantages over the usual viscose-making process, wherein sheets of pulp as such are steeped in caustic soda solution, as hereinbefore indicated. In such usual process, the sheets are of imperfect formation or texture and usually comprise localized zones or areas having greater density than other zones or areas. Sheets of such "lumpy" formation are inevitable, since the sheets are thick and are formed from substantially unbeaten pulp on high speed machines of the papermaking type. When the wet pulp sheet of "lumpy" formation is pressed at the wet end of the papermaking machine, it necessarily comprises high and low density localities; and the differences in density or texture in the sheet carry through to the finished or dried sheet delivered by the machine. The drier sheets hence cannot absorb caustic soda solution uniformly in the steeping press in which they are stacked in upstanding contactual relationship, for the denser or more compact portions of the sheets tend to take up less caustic soda solution per unit weight of pulp than the other portions. Although attempts have been made to improve the texture or uniformity and other qualities of pulp drier sheets, yet they are far from perfect as currently made and the producer of viscose must constantly compromise with the sheet qualities. Moreover, pulp drier sheets as commercially produced do not have uniform moisture content throughout their structure. The exposed or outer surfaces are usually overdried and their interiors under-dried in attaining the low moisture content commercially desired. By reason of the different degrees of dryness prevailing throughout the sheet structure, the fibers on the sheet surfaces and the fibers in the sheet interiors respond differently when immersed in caustic soda solution and they exhibit different degrees of reactivity when exposed to the carbon bisulphide. The result is that the viscose or cellulose xanthate solution prepared from such sheets really represents a blend of different kinds of cellulose xanthate rather than a product composed of uniformly xanthated cellulose. Inasmuch as the process hereof does away with the sheet form of cellulose preparatory to xanthation and the non-uniform characteristics of the cellulose incident to such sheet form, it makes possible an improvement in the quality of the resulting viscose or cellulose xanthate solution. Thus, in the process hereof the pulp fibers are resuspended or individualized in water and, after thickening, are substantially uniformly treated with the caustic soda solution to yield a substantially uniform pulp suspension in caustic soda solution in consequence of which the fibers are substantially uniformly xanthated when liquid carbon bisulphide is admixed with the suspension. The process hereof possesses the advantage over the one-step xanthating process of Richter application Serial No. 58,539, filed January 10, 1936, which involves the cutting of pulp-board or pulp drier sheets into chips prior to soaking and disintegration in caustic soda solution and subsequent xanthation, in that the cutting of pulp-board or drier sheets into chips tends to cause local compaction of the chips at their edges and, accordingly, resistance toward penetration of the caustic soda solution at such edges. The process hereof has another advantage over the usual viscose-making process in that slushing of the predried pulp in water and admixture of caustic soda solution with the thickened, slushed pulp suspension does not entail a removal of air from the pulp in securing uniform distribution of caustic soda solution throughout the pulp fibers, whereas, on the other hand, when drier sheets of pulp are steeped in caustic soda solution in the usual viscose-making process, difficulty is experienced in displacing the air present in the sheets by the caustic soda solution so as to attain the desired uniform infusion of caustic soda throughout the sheets. In other words, the thickened wet pulp produced in the process hereof is readily wetted uniformly throughout by caustic soda solution on account of its thoroughly wet condition and the tendency of the caustic soda solution to diffuse substantially uniformly therethrough.

We claim:

1. In a process of making viscose involving the admixture of cellulose fiber, caustic soda solution, and liquid carbon bisulphide and involving the use of cellulose fiber in the form of predried, aggregated fibers, those steps which comprise disintegrating the predried, aggregated cellulose

fibers in sufficient water to yield an aqueous fiber suspension of a fiber content less than 6% until a suspension of essentially only individualized fiber units is produced, thickening the resulting suspension to a fiber content of about 20% to 40%, admixing with the thickened wet fiber water and caustic soda in amounts to form a substantially individualized fiber suspension in caustic soda solution of a causticity and cellulose content each falling within a range of about 6% to 15%, and admixing liquid carbon bisulphide with the resulting fiber suspension in caustic soda solution to form viscose.

2. In a process of making viscose involving the admixture of cellulose fiber, caustic soda solution, and liquid carbon bisulphide and involving the use of wood pulp in the form of pulpboard, those steps which comprise disintegrating the pulpboard in sufficient water to yield an aqueous fiber suspension of a fiber content less than 6% until the pulpboard has been resolved into a suspension of essentially only individualized fiber units, dewatering the resulting suspension to form a thick, substantially non-fluent but thoroughly wet pulp mass of a water to pulp ratio greater than 1 to 1, admixing with the wet pulp mass water and caustic soda in amounts calculated to yield a substantially individualized pulp fiber suspension in caustic soda solution of a causticity and cellulose content each falling within a range of about 6% to 15%, and admixing liquid carbon bisulphide with the resulting suspension of pulp in said caustic soda solution to form viscose.

3. In a process of making viscose involving the admixture of cellulose fiber, caustic soda solution, and liquid carbon bisulphide and involving the use of cellulose fiber in the form of predried, aggregated fibers, those steps which comprise disintegrating the predried, aggregated cellulose fibers in sufficient water to yield an aqueous fiber suspension of a fiber content less than 6% until a suspension of essentially only individualized fiber units is produced, thickening the resulting suspension to a fiber content of about 20% to 40%, admixing with the thickened wet fiber a portion of strong caustic soda solution and then a portion of water in amounts calculated to form a substantially individualized fiber suspension in caustic soda solution of a causticity and cellulose content each falling within a range of about 6% to 15%, and admixing liquid carbon bisulphide with the resulting fiber suspension in caustic soda solution to form viscose.

4. In a process of making viscose involving the admixture of cellulose fiber, caustic soda solution, and liquid carbon bisulphide and involving the use of wood pulp in the form of predried pulpboard, those steps which comprise disintegrating the predried pulpboard in sufficient water containing a wetting agent dissolved therein to yield an aqueous fiber suspension of a fiber content less than 6% until the pulpboard has been resolved into a suspension of essentially only individualized fiber units, dewatering the resulting suspension to form a thick, substantially, non-fluent but thoroughly wet pulp mass of a water to pulp ratio greater than 1:1, admixing with the wet pulp mass water and caustic soda in amounts to form a substantially individualized pulp fiber suspension in caustic soda solution of a causticity and cellulose content each falling within a range of about 6% to 15%, and admixing liquid carbon bisulphide with the resulting suspension in caustic soda solution to form viscose.

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