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PROCESS OF PRODUCING VISCOSE

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This invention relates to a process of producing viscose or cellulose xanthate solution. Its objective, generally speaking, is to make for advantageous utilization of a dilute aqueous suspension of cellulose fiber, such as is available in a chemical wood pulp mill, in a one-step xanthating process, that is, a process involving the production of viscose or cellulose xanthate solution by the admixture all at once of cellulose fiber, caustic soda solution, and liquid carbon bisulphide.

In chemical wood pulp mills or pulp-refining mills, the finished wood pulp product is generally available as a dilute suspension of fibers, for instance, of about 2% to 4% fiber content, in the clean wash water used to remove the last chemical treating liquor, say, the bleach or anti-chlor liquor, applied to the pulp. The present invention makes use of such a dilute aqueous pulp suspension and involves thickening of the suspension to much higher fiber content or consistency, admixing concentrated caustic soda solution with the thickened suspension, and admixing liquid carbon bisulphide with the suspension of pulp in the resulting diluted caustic soda solution to form viscose or cellulose xanthate solution. The process hereof is advantageous in that it is a relatively simply and inexpensive matter to thicken a comparatively dilute pulp suspension with the machinery usually available in a pulp mill to much higher fiber content or consistency, say, about 20% to 40%, and then to reduce the fiber content of the suspension with caustic soda solution of suitable strength, for instance, a solution of 25% to 50% strength, in volume to provide a suspension in caustic soda solution of a strength or causticity, preferably not exceeding about 15%, appropriate for a one-step xanthating reaction. To the resulting suspension may be added liquid carbon bisulphide in suitable amount for forming the desired viscose or cellulose xanthate solution; and the mixed ingredients may be allowed to react to form the viscose or cellulose xanthate solution under the appropriate conditions, preferably with the aid of an organic liquid dissolved in the liquid carbon bisulphide and imparting to the carbon bisulphide solution a specific gravity approximating that of the caustic soda solution participating in the reaction, as set forth in detail in Richter and Lovering application Serial No. 153,688, filed July 14, 1937. Indeed, the various conditions of xanthation disclosed in that application may be maintained in the one-step xanthating reaction forming part of the process hereof.

Not only does the process hereof dispense with the drying of pulp and the expense incident thereto but the pulp, being used for the xanthating reaction in a form not yet once dehydrated or dried from the time of its liberation, is more readily reactive or xanthatable than wood pulp put up in the form of pulpboard or drier sheets and requiring considerable manipulation in water or caustic soda solution before the individual pulp fibers are segregated from one another so as to yield a fiber suspension in caustic soda solution substantially devoid of fiber bunches or aggregates, such as is desirable for a one-step xanthating reaction. In this connection, it is to be observed that the drying of pulp to produce pulpboard or drier sheets is accompanied not only by interfelting or firm integration of the pulp fibers but also by shrinkage of the fibers into a comparatively dense structure; and the manipulation in water of the dried sheets or pulpboard to induce the fiber-individualization and fiber-swelling desired for a one-step xanthating reaction accomplishes at best essentially nothing more than a partial restoration of the fibers to the favorable state for such reaction possessed thereby prior to drying. It is thus seen that, besides doing away with the steps of drying and reslushing pulp and the expense incident thereto, the invention hereof takes advantage of the originally individualized and reactive state presented by the pulp fibers in consequence of their being maintained wet or in aqueous suspension from the time of their liberation to the time of their participation in the one-step xanthating reaction.

The dilute aqueous suspension of cellulose pulp available for the purposes hereof may be wood pulp preliberated in any suitable way, for instance, in acid sulphite cooking liquor. Thus, sulphite wood pulp prepared by cooking chipped spruce or similar wood as ordinarily in an acid sulphite cooking liquor may be washed free of cooking liquor, bleached, washed, antichlored, and again washed to yield a 2% to 4% pulp suspension in clean water. Or the pulp suspension in clean water may be one of about 2% to 4% consistency eventuating from the treatment of washed, preliberated wood pulp in a succession of chemical liquors, including chlorinating liquor, alkaline refining liquor, bleach liquor, and antichlor liquor, each liquor treatment of the pulp being followed by washing of the pulp in water. The finished wood pulp in

dilute aqueous suspension as thus prepared may be of high whiteness and alpha cellulose content, for instance, an alpha cellulose content upwards of about 93%.

Starting with any one of the foregoing dilute aqueous pulp suspensions, the process hereof involves thickening the suspension to much higher consistency, for instance, from an initial fiber content of about 2% to 4% 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 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 caustic soda solution of proper strength, say, a caustic soda solution of 25% 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 strong 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.

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 the aforementioned Richter and Lovering application, 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 that 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.

It is to be understood that the particular numerical values hereinbefore cited are simply illustrative of certain conditions under which the process hereof may be performed and that the process hereof is not limited to the production of viscose or cellulose xanthate solution of a particular causticity and/or cellulose content, or to starting with a fiber suspension at a particular dilution, or to thickening the suspension to a particular consistency, or to diluting the thickened pulp with caustic soda solution of a particular strength, or to forming a fiber suspension of a particular fiber content and/or causticity for reaction. The essence of the instant invention resides in the new combination of steps involving the thickening to much higher fiber content of fiber available in the form of a prediluted aqueous fiber suspension, for instance, wood pulp as it emerges in diluted or washed aqueous suspension from a bleaching or other chemical treatment, the redilution of the thickened or wet pulp with strong caustic soda solution to provide a pulp suspension in caustic soda solution of a strength preferably less than about 15%, and the admixture of liquid carbon bisulphide in appropriate amount with the resulting suspension of pulp in caustic soda solution to form viscose. The prediluted aqueous fiber suspension may be so dilute as to be readily flowable as a stream to a rotary or other suitable thickening device. While aqueous fiber suspensions of about 2% to 4% fiber content are typical of the fluent fiber suspensions available for the purposes hereof, the invention is, of course, applicable to more dilute or fluent aqueous fiber suspensions, for instance, suspensions of a fiber content of about 1% or even less. In any event, such dilute or fluent fiber suspensions are thickened to much higher consistency preparatory for mixing or redilution with strong caustic soda solution, preferably to a consistency such that while the fiber is still very wet or unshrunken, it is substantially non-fluent and cannot be pumped or otherwise handled like water. The resulting fiber suspension in caustic soda solution is sufficiently fluent to enable the desired uniform mixing with the liquid carbon bisulphide. However, the suspension may be of a thick, paste-like consistency while being sufficiently fluent to permit uniform admixture therewith of the liquid carbon bisulphide.

We claim:

1. In a process of producing viscose for which is available a fluent aqueous suspension of chemically preliberated wood pulp maintained wet from the time of its preliberation, those steps which comprise thickening such pulp suspension to much higher fiber content while maintaining the pulp thoroughly wet, admixing concentrated caustic soda solution with the thickened wet pulp in volume to provide a fluent pulp suspension in caustic soda solution of a strength ranging from about 6% to 15%, and admixing liquid carbon bisulphide with the resulting fluent suspension of pulp in said caustic soda solution to form viscose.

2. In a process of producing viscose for which is available a fluent aqueous pulp suspension of chemically preliberated wood pulp maintained wet from the time of its preliberation, those steps which comprise thickening such pulp suspension to a substantially non-fluent state while maintaining the pulp fibers in thoroughly wet, unshrunken condition, admixing with the thickened wet pulp caustic soda solution of a strength and in an amount calculated to yield a pulp 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 fluent suspension of pulp in said caustic soda solution to form viscose.

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