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PROCESS FOR MANUFACTURING VISCOSE

Harold B. Vollrath, Bronxville, N. Y.

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My invention relates to the production of viscose and particularly to those steps in the process in which alkali cellulose is converted to cellulose xanthate and is subsequently dissolved or dispersed in alkali to form viscose.

In accordance with one method heretofore used in the manufacture of viscose, alkali cellulose in the form of "crumbs" is tumbled within a chamber while being treated with carbon bisulfide to convert the "crumbs" to cellulose xanthate. The excess, or unabsorbed, carbon bisulfide vapors are then drawn off and the resulting cellulose xanthate is removed to a dissolver where it is agitated or stirred with a solution of caustic alkali to dissolve it. The alkali is introduced in the form of a relatively dilute aqueous solution and the cellulose xanthate is added to it. The xanthating treatment ordinarily requires from 2 to 2½ hours; the dissolving operation requires 2 to 4 hours. Both of these steps are carried out at carefully controlled temperatures.

In transferring the xanthate to the dissolver considerable amounts of the carbon bisulfide vapors escape into the room in which the operations are conducted, endangering the health of the operators and presenting a serious fire and explosion hazard. Moreover, the xanthate upon exposure to air tends to convert to the undesirable oxycelluloses.

In accordance with another process, the steps of xanthating and dissolving are carried out in a single chamber. However, the material is kneaded rather than tumbled during the xanthating operation and a tough, rubbery mass is produced, which requires the application of considerable power and extremely careful technique to dissolve. During the dissolving operation the alkali is admitted to the chamber very slowly and in a relatively concentrated form and the water for dilution is subsequently added in a similarly controlled manner.

The power required to operate the kneading members in dissolving the material in this process is very great, the machine must consequently be built very strong and heavy and continual care is required in regulating and controlling the addition of alkali and water, for if either is added too rapidly, large lumps, or "flounders", are formed which cannot be dissolved except at an exorbitant consumption of time and energy, and the entire batch of material becomes worthless and must be discarded. The time required for completing the two operations of xanthating and dissolving by this method is greater than in the method previously described, and the expenditures encountered due to the power consumption and the losses of material due to occasional improper control in the dissolving operation are costly.

I have developed a method for the xanthation of alkali cellulose and the dissolving thereof to produce viscose which avoids the difficulties and hazards of previous methods and effects a substantial saving in capital investment, floor space, time power consumption, and labor. Moreover, my process eliminates the fire and explosion dangers of the process first described above while avoiding the high power consumption and losses due to improper control of the dissolving operation in the alternative process referred to above.

In accordance with a preferred method of procedure embodying my invention, alkali cellulose in comminuted form such as the usual "crumbs" is charged into a converter which may be of any suitable type, but which preferably is of the type described in my co-pending application Serial No. 83,201 filed June 3, 1936. After charging the converter with the alkali cellulose "crumbs" vacuum is applied to liberate entrapped or occluded air and the greater part of the air in the converter. The material may be stirred quietly during evacuation, if desired to liberate air from the entire batch of material to be treated. The carbon bisulfide subsequently introduced, therefore, does not have to displace air in and about the "crumbs" and within the converter in order to reach the material being treated and in order that it may pass rapidly and effectively into intimate contact with the alkali cellulose. In this way the reaction is hastened and substantially complete conversion of the alkali cellulose to cellulose xanthate assured.

After evacuation of the material the "crumbs" are treated with carbon bisulfide and are stirred slowly and gently in both the vertical and horizontal planes. During the treatment the "crumbs", therefore, are not only agitated gently but are raised and permitted to fall downwardly within the converter thereby preventing the formation of large lumps or masses and exposing the "crumbs" on all sides to the action of the carbon bisulfide vapors. The time and power required for effecting complete conversion of the alkali cellulose to cellulose xanthate is thereby reduced and kneading of the material with the resulting formation of large, tough, rubbery masses is avoided. The amount of carbon bisulfide used may be the same as that ordinarily used in the xanthation of alkali cellulose, about 33% of the weight of the dry cellulose, but the approximately 2% of CS_2 unabsorbed need not be

evacuated from the chamber after completion of the xanthating treatment and before dissolving. This excess CS_2, always required by the character of this organic reaction, will be largely dissolved in the viscose formed in the dissolving operation. The xanthating operation generally requires about 1½ to 2 hours and is carried out at the customary temperature.

After completion of the xanthating operation, the cellulose xanthate, which largely remains in the form of "crumbs" and is in a loose condition, is treated directly in the same chamber with the full amount of alkali solution required to produce a viscose of the desired alkali and cellulose content. Careful control and regulation of the concentration and rate of introduction of the alkali is not necessary. The entire quantity of alkali required for producing a final product of the desired alkali and cellulose content may be introduced rapidly upon starting the dissolving operation. However, if preferred, the alkali may be added otherwise, either slowly or in varying concentrations, as by introducing a relatively concentrated solution of alkali at first, with subsequent introduction of sufficient water or diluted alkali to insure proper concentration of cellulose and alkali in the final product.

Grinding, stirring, and churning of the xanthate suspended or floating in the alkali within the converter is then effected by suitable means. When using the mechanism of my co-pending application referred to above, the agitator which is rotated in one direction slowly during the xanthating operation, may be reversed and driven rapidly to grind and churn the cellulose xanthate and alkali solution together. Also, if preferred, the liquid carrying the xanthate therewith may be circulated by a pump or otherwise through a high speed grinding device in which the xanthate is further ground and churned. The fineness to which the material is ground in the latter device may be progressively increased as the operation continues, or the speed of the grinding members may be varied. Relatively small quantities of the mixture may thus be continuously drawn off and treated and the ground and churned mixture returned to the converter, while churning the remaining contents without interruption.

Cooling or control of the temperature of treatment both during the xanthating and the dissolving operations may be carried out in any suitable manner and the grinding and pumping equipment may be cooled if desired. Generally, the dissolving operation only requires about two hours, after which the batch may be discharged and the converter washed and made ready for the succeeding batch. However, the length of time required for dissolving or dispersing the xanthate will vary with the type of mechanism employed and the conditions of operation.

While I prefer to use the apparatus of my co-pending application in the practice of my invention, any suitable apparatus capable of performing the operations described may be used. Furthermore, the operations themselves may be varied considerably and the length of time, concentration of alkali and size of the batch treated may be altered as desired to adapt my invention to any particular manufacturing conditions. It should therefore be understood that the operations and conditions described are intended to be illustrative of typical procedure in accordance with my invention and are not intended to limit the scope of the following claims.

I claim:

1. In the manufacture of viscose the steps of charging alkali cellulose crumbs into a chamber, treating the crumbs with carbon bisulfide while stirring the crumbs slowly in both vertical and horizontal planes and maintaining the resulting cellulose xanthate in said chamber in the form of crumbs and in a loose condition free from large lumps or masses, adding to the resulting loose cellulose xanthate in said chamber the full amount of alkali solution required to produce a viscose of the desired alkali and cellulose content and agitating the mixture.

2. In the manufacture of viscose the steps of charging alkali cellulose crumbs into a chamber, evacuating the chamber to remove air and gas from the chamber and from the alkali cellulose, treating the same with carbon bisulfide while stirring the material in both vertical and horizontal planes and maintaining the resulting cellulose xanthate in the chamber in the form of crumbs and in a loose condition free from large lumps or masses, adding to the cellulose xanthate in said chamber the full amount of alkali solution required to produce a viscose of the desired alkali and cellulose content and agitating the mixture.

3. In the manufacture of viscose the steps comprising charging alkali cellulose crumbs into a closed chamber, treating the alkali cellulose crumbs with carbon bisulfide while stirring the same and maintaining the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the xanthate in said chamber during an initial stage of the dissolving operation substantially the entire amount of alkali solution required to produce a viscose of the desired cellulose and alkali content and agitating the mixture.

4. In the manufacture of viscose the steps of charging alkali cellulose crumbs into a closed chamber, introducing carbon bisulfide into the chamber while stirring the crumbs in such a way as to cause the crumbs to be raised and allowed to fall downwardly within the chamber and to maintain the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the resulting loose cellulose xanthate in said chamber during an initial stage of the dissolving operation substantially all of the alkali solution required for producing a viscose of the desired cellulose and alkali content and agitating the mixture.

5. In the manufacture of viscose the steps of treating alkali cellulose crumbs in a closed chamber with carbon bisulfide to produce cellulose xanthate while maintaining the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the material in said chamber the full amount of alkali solution required to produce a viscose of the desired alkali and cellulose content, and agitating the mixture vigorously while subjecting a portion of the mixture to still more intense comminuting and churning treatment.

6. In the manufacture of viscose the steps of treating alkali cellulose crumbs in a closed chamber with carbon bisulfide to produce cellulose xanthate while maintaining the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the material in said chamber the full amount of alkali solution required to produce a viscose of the desired alkali and cellulose content, and agitating the mixture vigorously while continuously subjecting successive portions of the

mixture to still more intense comminuting and churning treatment.

7. In the manufacture of viscose the steps of treating alkali cellulose crumbs in a closed chamber with carbon bisulfide to produce cellulose xanthate while maintaining the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding an alkali solution to the material in said chamber, agitating the mixture, withdrawing a small portion of said mixture from said chamber, subjecting said portion to more intense grinding and churning treatment and returning the ground and churned portion to said chamber.

8. In the manufacture of viscose the steps comprising charging comminuted alkali cellulose crumbs into a closed chamber, evacuating said chamber to remove air and entrapped gas from the alkali cellulose and from the chamber, treating the comminuted material with carbon bisulfide while stirring the same and maintaining the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the xanthate in said chamber during an initial stage of the dissolving operation substantially the entire amount of alkali solution required to produce a viscose of the desired cellulose and alkali content and vigorously agitating the mixture, while successively subjecting portions of the mixture to still more intense comminuting and churning treatment.

9. In the manufacture of viscose the steps of charging alkali cellulose crumbs into a closed chamber, evacuating said chamber, introducing carbon bisulfide into the chamber while agitating the crumbs in such a way that they are raised and allow to fall downwardly within the chamber and are maintained in the form of crumbs and in a loose condition free from large lumps or masses, adding to the resulting loose mass of cellulose xanthate in said chamber during an initial stage of the dissolving operation substantially all of the alkali solution required for producing a viscose of the desired cellulose and alkali content and vigorously agitating the mixture, while continuously withdrawing portions of said mixture from said chamber and subjecting the same to still more intense grinding and churning treatment and returning the ground and churned portions to said chamber.

10. In the manufacture of viscose in a chamber having stirring means therein the process steps of charging alkali cellulose crumbs into said chamber, introducing carbon bisulfide into said chamber while actuating said stirring means in such a way as to maintain the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the resulting loose cellulose xanthate in said chamber an amount of alkali solution sufficient to produce a viscose of the desired alkali and cellulose content and actuating said stirring means in such a way as to grind and churn the cellulose xanthate in the alkali solution to produce viscose.

11. In the manufacture of viscose in a chamber having stirring means therein the process steps of introducing alkali cellulose crumbs into said chamber, closing said chamber, treating said alkali cellulose crumbs with carbon bisulfide while actuating said stirring means in such a way as to maintain the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the material in said chamber the full amount of alkali solution required to produce a viscose of the desired alkali and cellulose content, actuating said stirring means in such a way as to grind and churn the cellulose xanthate in the presence of the alkali solution and successively subjecting portions of the mixture to still more intense comminuting and churning treatment within said chamber.

12. In the manufacture of viscose in a chamber having stirring means therein the process steps of charging alkali cellulose crumbs into said chamber, closing said chamber, evactuating said chamber to remove air and entrapped gas from the alkali cellulose and from the chamber, introducing carbon bisulfide into said chamber and into contact with the alkali cellulose crumbs, actuating said stirring means slowly and in such a way as to maintain the resulting cellulose xanthate in the form of crumbs and in a loose condition free from large lumps or masses, adding to the xanthate in said chamber during an initial stage of the dissolving operation substantially the entire amount of alkali solution required to produce a viscose of the desired alkali and cellulose content, thereafter actuating said stirring means in a different manner and in such a way as to grind and churn the cellulose xanthate in the presence of the alkali solution and successively subjecting portions of the mixture to still more intense comminuting and churning treatment within said chamber.

HAROLD B. VOLLRATH.