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PRODUCTION OF VISCOSE

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This invention relates to improvements in methods of producing viscose.

It is an object of the invention to provide a method for producing viscose of low alkali content which is free from undissolved or incompletely dissolved fibrous lint and which is readily amenable to filtration and spinning.

It is another object of the invention to provide an improved method involving a multi-stage procedure whereby an intermediate viscose of relatively high alkali content is first produced which is adapted to be subsequently brought down to the weak final or terminal concentration desired.

To this end the procedure contemplates the satisfactory dissolution of the xanthate by subdividing the production of the lean viscose into two stages, one involving a high alkali content for completely dissolving the xanthate and the other a dilution stage for bringing the concentrated viscose to the proper terminal dilution, the two stages being separated by a rest period, as hereinafter described.

During recent years it has become quite customary in the artificial thread industry to work up viscose varieties which, considered in proportion to their cellulose content contain but small quantities of alkali and which therefore are cheaper and also require less sulphuric acid during spinning—examples thereof being viscoses having 8% cellulose and 5% alkali or 7% cellulose and 4.5% alkali (NaOH) content. Viscoses which were prepared in essentially the same manner as ordinary viscoses containing 8% cellulose and 7% NaOH content or 7% cellulose and 7% alkali content—that is, as made by dissolving the xanthate in the calculated quantity of a mixture of caustic soda and water, occasioned appreciable difficulties during the processing, both during filtration and during the spinning operations, since they always contained a relatively large amount of undissolved or incompletely dissolved broken pieces of fibres or fibrous lint.

It has however been found that in the production of weakly alkaline or so-called "lean" viscose, it is erroneous during the dissolution of the xanthate into viscose to predicate the control of the procedure directly upon the final composition of the weakly alkaline viscose, but that instead, it is preferable to produce an intermediate viscose with a high alkali content of about 9 to 10% and above, and then to dilute this viscose subsequently with water or diluted caustic soda to the final composition desired. Tests have shown, for example, that the filtration difficulties and poor spinning properties of the weakly alkaline or lean viscose as heretofore produced are not due to the circumstance that the prevailing specific absolute concentration of caustic soda in the viscose is not capable of maintaining the cellulose or cellulose xanthate in a perfect dissolved condition, but that the fault rather lies in that the xanthate (in the prior art method of procedure) is practically never satisfactorily dissolved and that a satisfactory solution is possible if during the dissolution thereof an intermediate stage be first created wherein the viscose dilution is adjusted to over 8% and preferably 9% and above of alkali content and if it be maintained for a certain time period, say, for example, 15, 30, or 60 minutes; and it has been found that thereafter such a well dissolved viscose, initially rich in alkali, can be diluted without danger with the most dilute caustic soda or even water to achieve the weakly alkaline final composition desired.

The essence of the present invention is therefore to subdivide the entire production of a lean viscose (or viscose of weak alkali concentration) into two stages so as first to produce a viscose of high cellulose and alkali content, i. e., an alkali content in excess of 8% and preferably of at least about 9%, and then, after a rest period and in a second operation to dilute this concentrated viscose with very dilute caustic soda or even water to produce the final composition desired. The success of this method finally depends (as has also been found) on the fact that it is only the higher alkali concentrations of 9, 10, 12, 15% and above which are capable of satisfactorily dissolving the cellulose xanthate, i. e., even the last traces of xanthated fibers.

The method may with advantage be practised with the aid of a so-called "vacuum xanthate kneader" in which both the xanthating and dissolving operations may be performed. After the first stage of the dissolution procedure has been carried out with the addition of water or diluted liquor until a viscose slurry concentration of about 9% alkali content is obtained, further addition of diluted liquor or dilution with water is temporarily suspended, the stirrer being allowed to continue running, and the viscose mass is given time to pass into solution under the action of the 9% liquor concentration and it is only after this is effected that further dilute caustic soda or solvent water is added thereto to produce the final concentration desired. The time lost, during which additional dilution water must not be added is abundantly compensated by the fact that the viscose can thereafter be very readily diluted to the final composition desired.

The xanthate passes most easily and most completely into solution in the 9 and 10% range of alkali concentration and the essence of the invention consists in also utilizing this observation for the production of weakly alkaline viscose. An illustrative embodiment of the method of practising the invention is given herewith.

Example

Cellulose of a type conventionally used in the rayon industry is dipped in conventional manner in caustic soda, pressed, shredded, preliminarily ripened and xanthated. The cellulose xanthate is then first kneaded in a suitable kneader with a suitable quantity of caustic soda so diluted as to result in the production of a viscose composition of about 16% cellulose and 9% NaOH content. This final composition contains about 4–5% of alkali and about 7–8% of cellulose by weight. For this purpose the caustic soda is added gradually as the kneading and stirring proceed. After the addition is effected, the stirrer is allowed to continue running for one hour in order to insure thorough dissolution, this interval being regarded as a rest period. Thereupon, in a second working stage, the remaining solvent water is likewise gradually added, with continued stirring, and in such an amount as to produce the desired final alkali concentration of 4.5% for an 8% cellulose content. The viscose obtained, when examined under the microscope, is substantially devoid of free fibres and presents but little difficulty during filtration and spinning.

Modifications of this invention will readily be recognized by those skilled in the art, and it is desired to include all modifications and variations coming within the scope of the appended claims.

What is claimed is:

1. In the process of producing viscose solutions poor in alkali and devoid of free fibres, the steps which comprise in sequence forming an initial viscose solution having an alkali content in excess of 8% and a cellulose content of about 16% by weight, allowing said solution to rest for a period of about 15 to 60 minutes, and diluting said initial viscose solution to form a final viscose solution having an alkali content of about 4 to 5% and a cellulose content of about 7 to 8% by weight.

2. In the process of producing viscose solutions poor in alkali and devoid of free fibres, the steps which comprise in sequence forming an initial viscose solution having an alkali content in excess of 8% and a cellulose content of about 16% by weight, allowing said solution to rest for a period of about 15 to 60 minutes, and diluting said initial viscose solution with a sodium hydroxide solution to form a final viscose solution having an alkali content of about 4 to 5% and a cellulose content of about 7 to 8% by weight.

3. In the process of producing viscose solutions poor in alkali and substantially devoid of free fibres, the steps which comprise in sequence forming an initial viscose solution having an alkali content of about 9 to 10% and a cellulose content of about 16% by weight, allowing said solution to rest for a period of about 15 to 60 minutes, and diluting said initial viscose solution with water to form a final viscose solution having an alkali content of about 4 to 5% and a cellulose content of about 7 to 8% by weight.

4. In the process of producing viscose solutions poor in alkali, and substantially devoid of free fibres, the steps which comprise in sequence forming an initial viscose solution having a sodium hydroxide content of about 9% and a cellulose content of about 16% by weight, allowing said solution to rest for a period of about 60 minutes, and diluting said initial viscose solution with water to form a final viscose solution having a sodium hydroxide content of about 4.5% and a cellulose content of about 8% by weight.

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