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CUPRAMMONIUM CELLULOSE SPINNING PROCESS

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5 Claims. (Cl. 18—54)

In the process of producing shaped articles like bands, films and especially artificial fibers, from cuprammonium cellulose solutions according to the stretch-spinning method, large quantities of water are necessary as precipitating agent. For this purpose, the water is to be of very constant composition and very high purity. In order not to have to prepare such large quantities of fresh water of the required standards, it has been proposed to re-use the water having been employed in precipitation for precipitating further quantities of the cellulose solution. However, these processes did not give entire satisfaction. For instance, the coagulating action of water which has already been used is diminished because the water has taken up ammonia. In order to compensate this diminished action, for instance higher temperatures have been employed when precipitating further quantities of cellulose solution by means of such water. This resulted in clogging of the pipes and spinning funnels by particles of cellulose and copper hydroxide or oxide separating from the water, thereby causing considerable disturbances of the spinning process. It has also been tried to free the precipitation water from copper and/or ammonia and thereupon to re-use it in precipitation. However, this necessitates complicated and expensive processes and apparatus for the recovery of the said constituents, especially because the same are present in very small quantities.

It is an object of our invention to obviate these difficulties and to furnish a simple and effective method of re-using the precipitation-water in the process of stretch-spinning cuprammonium cellulose solutions. We have found that the precipitation-water in such processes can be re-used without difficulty after it has been subjected to an intensive filtration. This may be effected, for instance, by means of a sand filter; the sand may also be replaced by kieselguhr or a similar material possessing a large active surface. Surprisingly, such filters do not only retain coarse particles, but also the above mentioned particles of cellulose and of copper compounds which are very finely (colloidally) dispersed in the water and which, in the non-filtrated water, give rise to the above mentioned agglomerations. According to our process, also these particles are practically completely removed from the water, so that the previous disturbances caused thereby are avoided. The water filtrated in this manner can be directly used for precipitating further quantities of cellulose solution, if desired after the addition of a suitable amount of fresh water.

A further advantage of re-using the water in precipitating cuprammonium cellulose solutions is in the fact that the ammonia and copper content of the water is increased so that the recovery of the said ingredients is facilitated. This advantage becomes especially apparent if the fresh water is used for spinning artificial fibers of low total titer and the water therefrom, after filtration, is employed for precipitating fibers of high total titer. When working in this manner, the water takes up relatively small amounts of ammonia and copper in the first stage, whereas in the second stage it comes into contact with a much larger quantity of cellulose solution and, therefore, takes up large amounts of ammonia and copper. For instance, artificial silk is spun having a total titer of 15–200 den. (per each spinning nozzle) with fresh water, and the water, after filtration, is used for spinning artificial fibers of a total titer of 1000–3000 den. per spinning nozzle (as usual in staple fiber production).

Of course, the coagulating action of the water which has been used already in precipitation is smaller than the action of fresh water. It can be compensated in known manner, for instance by raising the temperature. The water may be re-used once or several times, provided it is filtrated as above stated before each new use. Obviously this has an end when the ammonia content of the water has reached a certain degree, because then the water ceases to have a coagulating action on the cellulose solutions.

We claim:

1. The process of producing shaped cellulose articles, especially artificial fibers, from cuprammonium cellulose solutions by stretch-spinning which comprises spinning the cellulose solution into water as a precipitating agent, precipitating the solution with the water, filtering the water used in precipitating the solution in a filtering medium capable of removing the colloidal copper and cellulose in the water, leaving substantially all of the ammonia in the water, and re-using the filtered water containing the ammonia to precipitate further quantities of cellulose solutions.

2. The process as defined in claim 1 wherein the filtering medium is sand.

3. The process as defined in claim 1 wherein the filtering medium is kieselguhr.

4. The process as defined in claim 1 wherein the water is first used in the precipitation of spun artificial fibers of low total titer and after filtration is used to precipitate spun artificial fibers of high total titer.

5. The process as defined in claim 1 wherein the water is originally used to precipitate spun artificial fibers having a titer of from 15 to 200 den. and the water after filtration is used to precipitate spun artificial fibers of a total titer of 1,000 to 3,000 den.

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