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## MANUFACTURE OF ARTIFICIAL VISCOSE PRODUCTS

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1 Claim. (Cl. 18—54)

This invention relates to a new and improved method for the manufacture of artificial viscose products employing zinc compounds in a coagulating bath and an acid bath for the after-treatment.

It is well known that with such a method, especially in the manufacture of artificial threads of viscose rayon, an unpleasant phenomenon occurs in the after-treatment baths manifesting itself in the separation of zinc sulphide which apparently first deposits in a sticky state on the sides of the after-treatment devices and on the conveying members, and thereafter forming crusts. Such crusts necessitate repeated cleansing of the devices and other apparatus elements mentioned. Moreover, during the manufacture of the threads of viscose rayon, they also frequently give rise to the formation of broken filaments which wind themselves up on the conveying and guiding devices and cause thread breakage and otherwise are troublesome and detrimental to the smooth operation of the overall process.

Many experiments have been made to investigate the circumstances under which the above mentioned unpleasant phenomena occur and how they might be prevented. It was found that the separation and deposition of zinc sulphide, irrespective of the presence of zinc compounds and hydrogen sulphide, depend on the acidity of the after-treatment bath concerned. Thus, it was discovered that the separation and deposition of zinc sulphide may begin when the after-treatment bath has a low acid concentration, e. g. 50 g. of  $H_2SO_4$  per litre, and that separation and deposition of the zinc sulphide are practically certain to occur when the acid concentration falls much below this value, for example, when it drops to about 30 g. of  $H_2SO_4$  per litre.

It is therefore an object of the present invention to provide a new and improved method whereby the separation and deposition of zinc sulphide can be prevented under all circumstances likely to be encountered in the manufacture of artificial viscose products, and especially viscose rayon threads.

The manner in which this and other objects of the present invention are attained will become apparent from the following description of the invention which is intended to be illustrative rather than limitative.

According to the present invention, it has been discovered that the undesired separation and deposition of zinc sulphide may be prevented by adding sufficient formaldehyde to the acid after-treatment baths, still containing zinc ions, to bind at least the greater part if indeed not all of the hydrogen sulphide, but no more than 2 mols of formaldehyde per mol of hydrogen sulphide present. By "hydrogen sulphide present," which must be con-

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tinuously bound, is meant the sum of the amount of hydrogen sulphide that is set free in the given bath by the action of acid on the product (e. g., thread) not yet fully decomposed, and the amount of hydrogen sulphide that may be introduced into the given bath by the product (e. g., thread) being processed and the liquid adhering to it.

It has already been proposed to add aldehydes, such as formaldehyde, to viscose spinning baths and also to after-treatment baths. Those proposals, however, relate to a modified "stenosage process." In the spinning baths employed, however, no zinc ions are present. Also, formaldehyde in alkaline medium is employed in very high concentrations, and finally, the spun yarn impregnated with formaldehyde is subjected to a special after-heating in order to bring about chemical reaction between the formaldehyde and the cellulose.

It has also been proposed in the manufacture of viscose rayon to add formaldehyde to the spinning bath or to the second bath to improve the stretching process. However, the formaldehyde concentrations proposed for that purpose are very much higher than those according to the present invention which are employed in order to prevent the separation and deposition of zinc sulphide, and which concentrations indeed exert no discernible influence on the stretching process.

When employing the method according to the present invention, it is desirable to check the addition of formaldehyde to the after-treatment bath by determining the formaldehyde content of this bath. This checking of the formaldehyde content may be carried out continuously or at predetermined intervals. Good results are obtained, especially in the manufacture of viscose rayon of high denier suitable for the manufacture of automobile tire cord, when the formaldehyde concentration in the after-treatment bath is maintained at about 0.05 to 0.2% by weight.

In order still further to illustrate the present invention, the following example of procedure in accordance with the invention is set forth by way of description.

### Example

Ripened viscose ready for spinning, and containing 7.7% of cellulose and 7.1% of NaOH, was spun in a spinning bath at 48° C. having a concentration (per litre) of 100 g. of sulphuric acid, 175 g. of sodium sulphate, 50 g. of magnesium sulphate and 30 g. of zinc sulphate. The viscose was spun to rayon thread of high denier consisting of 700 filaments. Immediately thereafter, the freshly spun thread was led through an after-treatment bath maintained at 80° C. and having a content (per litre) of 15 g. of sulphuric acid, 1.2 g. of formaldehyde, and the usual metallic salts. In this bath the thread was stretched by about 78%. The resulting thread of 1700 denier was wound into a centrifuge and further treated.

After 5 days of continuous spinning in this fashion, the total precipitate of zinc sulphide was collected and was found to be a bare  $\frac{1}{10}$  part of the amount of zinc sulphide that was otherwise formed under the same conditions, but without the addition of formaldehyde to the after-treatment acid bath.

While a specific example of a preferred method embodying the present invention has been described above, it will be apparent that many changes and modifications might be made therein without departing from the spirit of the invention. It will therefore be understood that the

particular procedure set forth above is intended to be illustrative only and not as limiting the invention.

What is claimed is:

In a method of manufacturing high denier viscose rayon yarn in which a two-bath system is used and the viscose is spun into a coagulating bath containing zinc sulphate, and the resulting yarn is passed to a second hot sulphuric acid-containing bath and during passage therethrough the yarn is stretched, the improvement which comprises add-

ing about 0.05% to 0.2% by weight of formaldehyde to the second bath which is sufficient to bind the greater portion of hydrogen sulphide present but insufficient to exert any discernible influence on the stretching of the yarn.

References Cited in the file of this patent

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